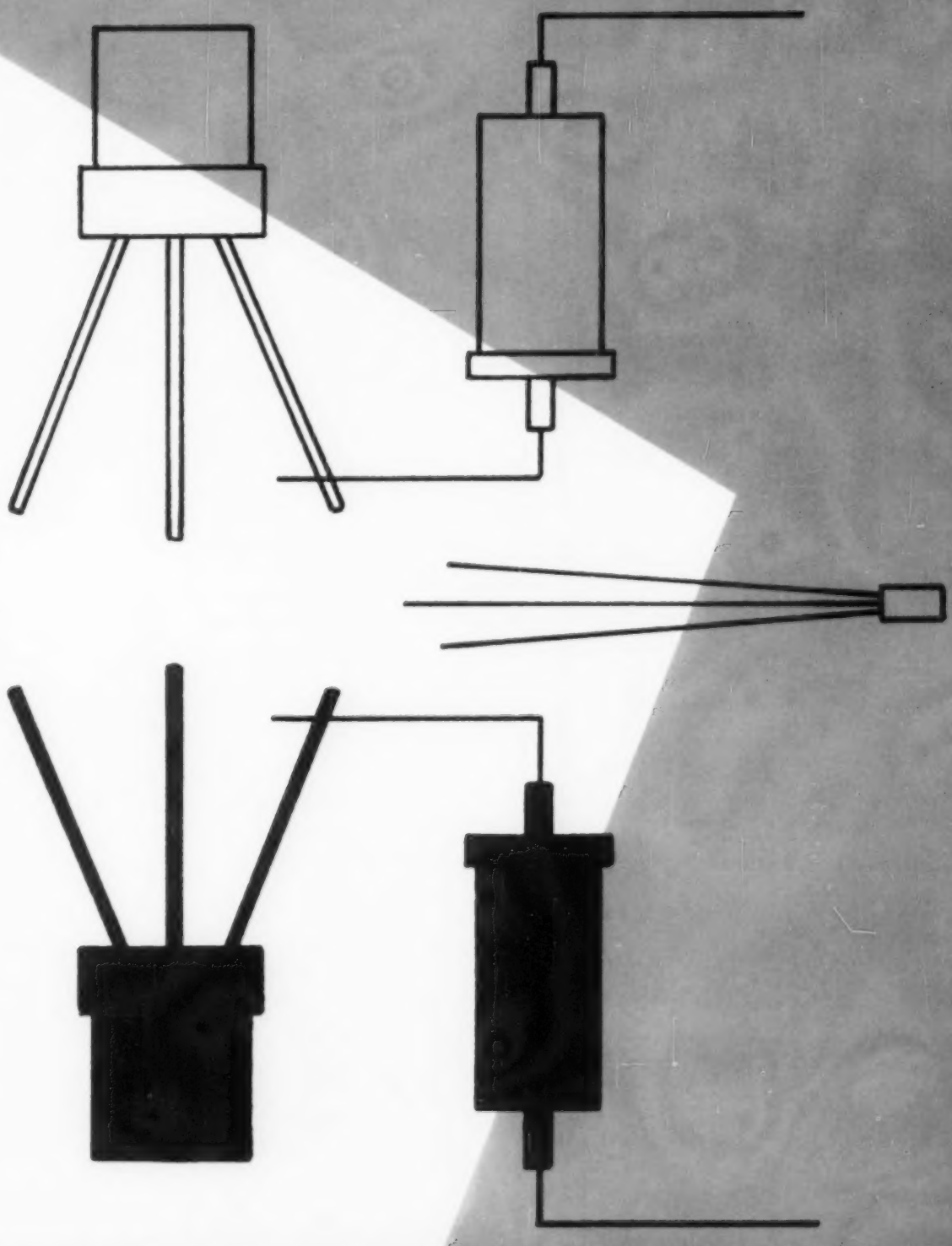
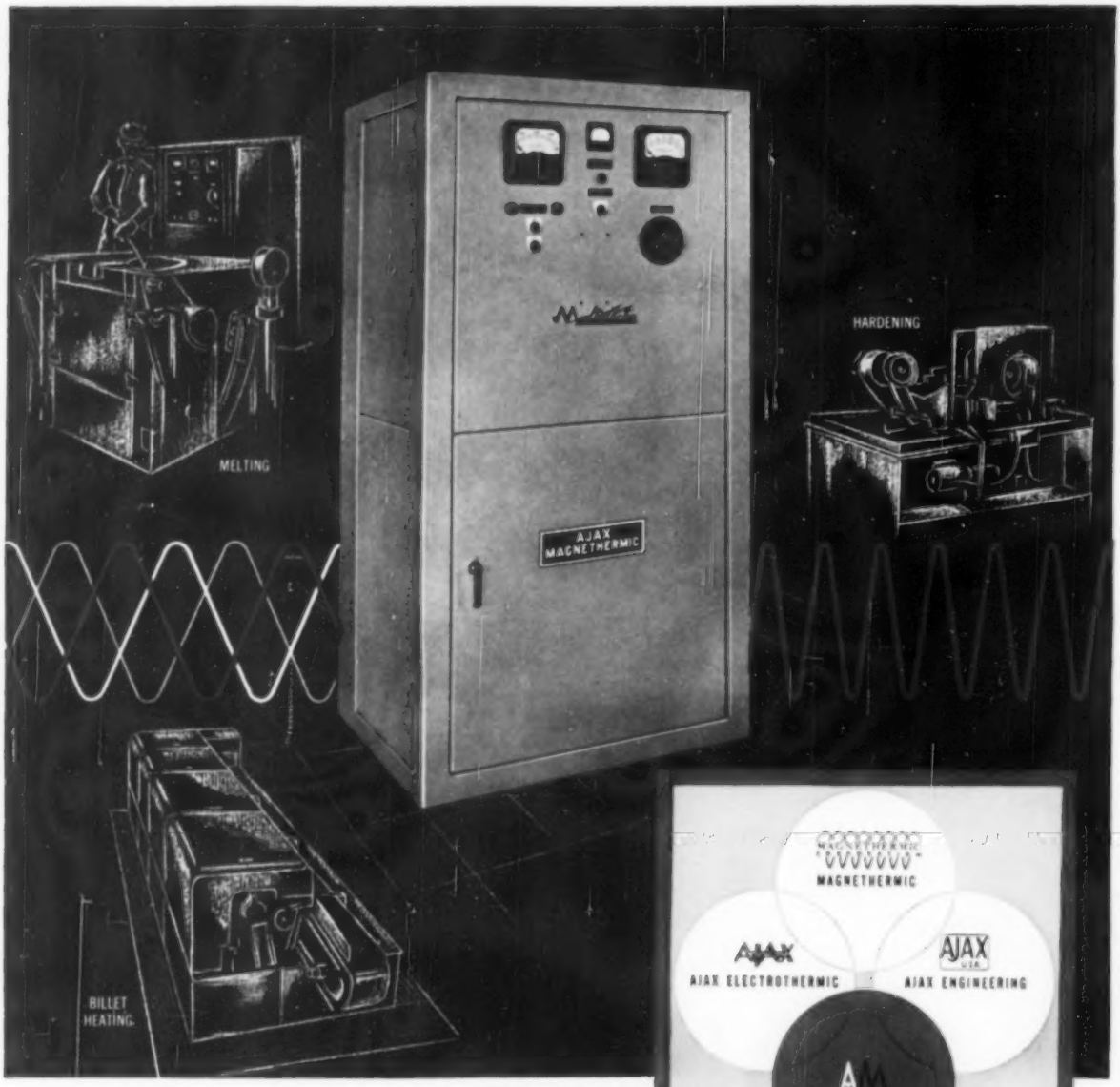
JOURNAL OF

METALS

NOVEMBER 1960





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JOURNAL OF

METALS

Vol. 12, No. 11
November, 1960

COVER: Looking over literature on semiconductors, our artist, David Cain, produced this month's cover which is dedicated to the IMD Semiconductors Committee. The story of this committee is told on the editorial page, while additional information is found on pages 837 and 875.

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personnel

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—MEN AVAILABLE—

All those listed under "Men Available" have membership in the AIME.

Metallurgical Engineer, B.S., Met. Eng., age 33. Eight years in aluminum industry in research and development on weldable alloys; three years in production of wire bar and sheet. Available immediately. Prefer northeast or west. M-1175-Chicago.

Chief Metallurgist-Mill Manager, B.S., Met. Eng., age 41. Twelve years experience as ore dressing metallurgist, research-development, plant operation and administration. Broad experience with complex ores, flotation, roasting, leaching, etc. Willing to travel but must be in good living and educational area. Prefer North America or British Commonwealth. M-1180-Chicago.

Technical Assistant to executive officer of metallurgical company, bachelor's and doctorate degrees. Eleven years physical and powder metallurgy, research and production. Familiar with technical and economic evaluation of development projects including preparation of sales and financial projections and appraisal reports for study at corporate level. Prefer New York metropolitan area. M-245.

Metallurgist, 25. One year experience as mineral dressing technician. Two years metallurgy and ceramic development, metals and metallic hardware problems, uranium reduction. Interested in private industry. $8400. Prefer west coast, any. Home: California. Se-1641.

Plant Supervisor, 52. Over twenty years experience supervision of anodizing and all metal finishing problems, for electromechanical products, automotive parts, bearings, plating equipment and solutions. $10,200. Prefer midwest, northwest. Home: Michigan. Se-1523.

A weekly bulletin of engineering positions open is available to AIME members at a subscription rate of $4.50 per quarter or $14 per annum; $4.50 per quarter or $14 per annum for nonmembers, payable in advance. Local offices of the Personnel Service are at 8 W. 40th St., New York 18; 57 Post St., San Francisco; and 29 E. Madison St., Chicago 1.

Industrial Engineer, 45, over twenty years experience supervision of I.E. departments, seventeen years installation, direct, indirect, and supervisor, incentive plans, cost improvement programs, equipment and facility evaluation analysis, production and inventory controls, method improvements, waste and scrap studies, and layout. Experienced heavy and precision machining, foundry, die casting, complex electromedical apparatus, and various assembly operations. Desire San Francisco area. $9000. Home: California. Se-1720.

—POSITIONS OPEN—

Physical Metallurgist, B.S. degree. a) Interest in metallography, to undertake specimen preparation and photomicrography in a research laboratory dealing with non-ferrous metals. b) To work on casting, fabrication and heat treatment of experimental alloys. c) To work on metallurgical problems associated with X-ray fluorescence analysis. Some experience in fluorescence analysis and familiarity with X-ray diffraction theory highly desirable. d) For elevated temperature tensile testing and eventual work in fatigue, creep and stress rupture. e) Knowledge of welding techniques, for research and development on welding of non-ferrous metals. f) Knowledge of electro-chemistry for basic research on corrosion problems of non-ferrous metals. Connecticut. W9626.

Metallurgist or Chemical Engineer, advanced degree preferred but recent applicable experience most valuable. Should have experience in some of the following fields: high temperature metals and ceramics, joining of metals and ceramics, vacuum deposition of metals, combustion processes; for project involving conversion of heat to electricity. Salary open. Connecticut. W9625.

Research Metallurgist, B.S. or M.S. in metallurgy, with experience in ferrous and/or non-ferrous metallography, phase diagram studies, mechanical testing, to conduct research and development work in alloy development and physical metallurgy. $8000-$11,000 year to start. Fee negotiable. Pennsylvania. W9615.

Jr. Chemical Engineer, graduate, with heavy chemical experience, to assist project engineer in selection and layout of metal ore processing equipment. $6000-$7200 year. Eastern Pennsylvania. W9598.

Metallurgist, for R&D, graduate, with three to five years experience, for development work on stainless steels, tool steels and general ferrous physical metallurgy. Applicant must be capable of planning and conducting experimental programs and writing clear, concise technical reports. Salary open. Pennsylvania. W9593.

Metallurgical Engineers, recent graduates or with up to five years experience, for development work with leading producer of ferrous, non-ferrous and reactive metals specialty tubing. Modern laboratory of multi-plant company. East. W9580.

Engineer, metal finishing, graduate chemical, with minimum of five years metal finishing experience. Preferably organic enameling, plating, anodizing and finishing problems in general; particularly dealing with small components. Maximum, $11,000 year. Pennsylvania. W9580(a).

Metallurgists experienced in powder metallurgy, vacuum metallurgy and special thermoelectrical materials. Eastern Canada. F9553.

Metallurgist, B.S. degree in metallurgical or chemical engineering, preferably with some related experience in concentration processes, particularly flotation and/or agglomeration. Will work in research laboratory or research pilot plant, on work chiefly related to concentration and agglomeration development on low grade iron ores. $6480-$7500 year. Michigan. W9538.

Job Engineer, engineering background, at least five years experience in general construction and engineering of buildings and concrete structures. Will work in the field and should be able to make building survey layouts, shop drawings, field changes and to work with superintendent in general contractor's office. About $7800. Northern California. SJ-5504.

Sales, Application, Commercial Engineers, B.S., M.S., M.E., E.E., Phys., ChE., Chem., or equivalent experience in specific areas covering one to ten years. Need sales-general manager, manager engineer or trainee, applications-manager; laboratory engineer; commercial-product or technical engineer. Will direct, manage, plan, supervise, conduct or assist in marketing, advertising, economic analysis, feasibility evaluation, promotion with customers and users, o.e.m.'s, government, representatives, distribution, dealers, and support programs at national, regional, or local levels to develop sales of electronic systems, devices, components, cryogenic products and equipment, communication, control and signalling devices and systems, power tubes, X-ray, resistors, semi-conductors, or tubes; including sales headquarters, office, field, liaison, literature, catalog. Principally non-military; for a major manufacturing corporate complex. Longtime growth record. $20,000, $15,000 plus, $12,000, $10,000, $8,000. New York area, New Jersey, Pennsylvania, Massachusetts. SJ-5515.

Metallurgical Engineer, Met., Chem, or MinE or equivalent. Basic knowledge of ore dressing methods and practices, and several years experience in minerals beneficiation. Involves working with small metallurgical engineering group at concentrate plants; process evaluation, evaluation of milling equipment and practices, various testing in connection with plant research and development in metallurgical process control and improvement. Salary open, opportunity for advancement in metallurgical activities or plant operations. West. SJ-5550.

Plant Engineer, Design, CE or archtl background, well experienced in site and office engineering, including drafting plans and designs, preferably with some knowledge of electrical and mechanical work. Should be able to gather information on program and costs for construction activity. Also estimate costs, make computations, prepare or interpret specifications. Must be able to make drawings for alterations, changes and new construction on buildings and structures, some of which may be tilt-up, concrete or precast. For manufacturing company. Santa Clara County, Calif. $7200-$7800. SJ-5494.

Teaching Personnel for Dept. of Mechanical Engineering, M.S. or Ph.D. required. a) Assistant or associate professor to teach thermodynamics and fluid mechanics. b) Assistant or associate professor to teach machine design and metallurgy. Salaries dependent upon qualifications. Midwest. W9582.

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Spokane 69, Wash.

METALGRAMS

UNION CARBIDE METALS

. . . news about "Electromet" ferroalloys and metals

NOVEMBER, 1960

AN EFFICIENT BLOCK -- Why is silicomanganese the most popular blocking alloy in the steel industry today? It dissolves 50 per cent faster than separate silicon and manganese additions, giving faster blocks and shorter blocking times. Its low-carbon content allows higher tapping carbons, reducing furnace time and also aiding steel cleanliness. Since silicomanganese is a more powerful deoxidizer than straight silicon blocks, it gives tighter blocks that last longer. Finally, silicomanganese provides low-cost silicon and manganese, reducing blocking costs to a minimum.

* * *

BETTER CONTROL, TOO! -- Results of hundreds of open-hearth production heats show that a silicomanganese block gives better control of carbon content and alloy additions. Off-grade heats for carbon and manganese have been shown to be lowest for silicomanganese compared to a pig ferrosilicon or a 50 per cent ferrosilicon block. Also, silicomanganese gave a higher manganese recovery than other blocks when all the manganese was added to the furnace. For details, write for the article, "Silicomanganese Improves Blocking Performance," in the Fall 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

LOWER COSTS FOR STAINLESS STEEL -- Another manganese-silicon alloy -- ferromanganese-silicon -- is saving stainless steel producers thousands of dollars per year. Used for slag reduction and manganese additions, ferromanganese-silicon is replacing other low-carbon manganese sources. Savings per ton: $0.75 to $1.75 for type 300 stainless grades and $4 to $9 for type 200 grades. Some stainless producers have also found that ferromanganese-silicon produces cleaner steels. Write for F-20,093 for further information.

* * *

BUILDING BIGGER MARKETS -- "Your man's practically a member of my staff," a president of a steel firm said of one of UCM's market development specialists. These men work with steel producers to develop new markets and expand old ones. Primarily, they promote the use of stainless steel and titanium (including castings) in transportation, architecture, process industries, and consumer goods. More details can be obtained by writing for the article, "Men, Metals, and Markets," in the Spring 1960 issue of UNION CARBIDE METALS REVIEW.

* * *

PRODUCTS WITH A SPLIT PERSONALITY -- Several "Electromet" alloys and metals are used in both metals and chemicals. Silicon metal -- an important alloy in aluminum and copper -- is a key raw material for silicones. Vanadium oxide -- used in tool and constructional steels -- is a catalyst in the production of adipic acid, sulfuric acid, maleic anhydride, and phthalic anhydride. It also gives special colors to glass and ceramics. Finally, "Elmang" electrolytic manganese -- high-purity manganese for stainless steel, deep-drawing steels, copper and aluminum -- is a catalyst for adipic, terephthalic, and acetic acids. Write for the article, "Products with a Split Personality," in the Spring issue of UNION CARBIDE METALS REVIEW.

* * *

UNION CARBIDE METALS COMPANY, Division of Union Carbide Corporation, 270 Park Ave., New York 17, N. Y. In Canada: Union Carbide Canada Ltd., Toronto.

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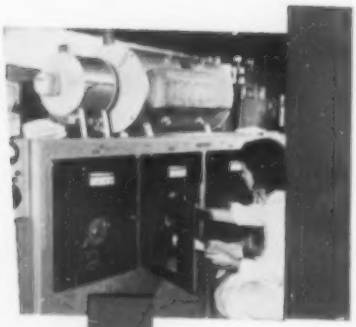

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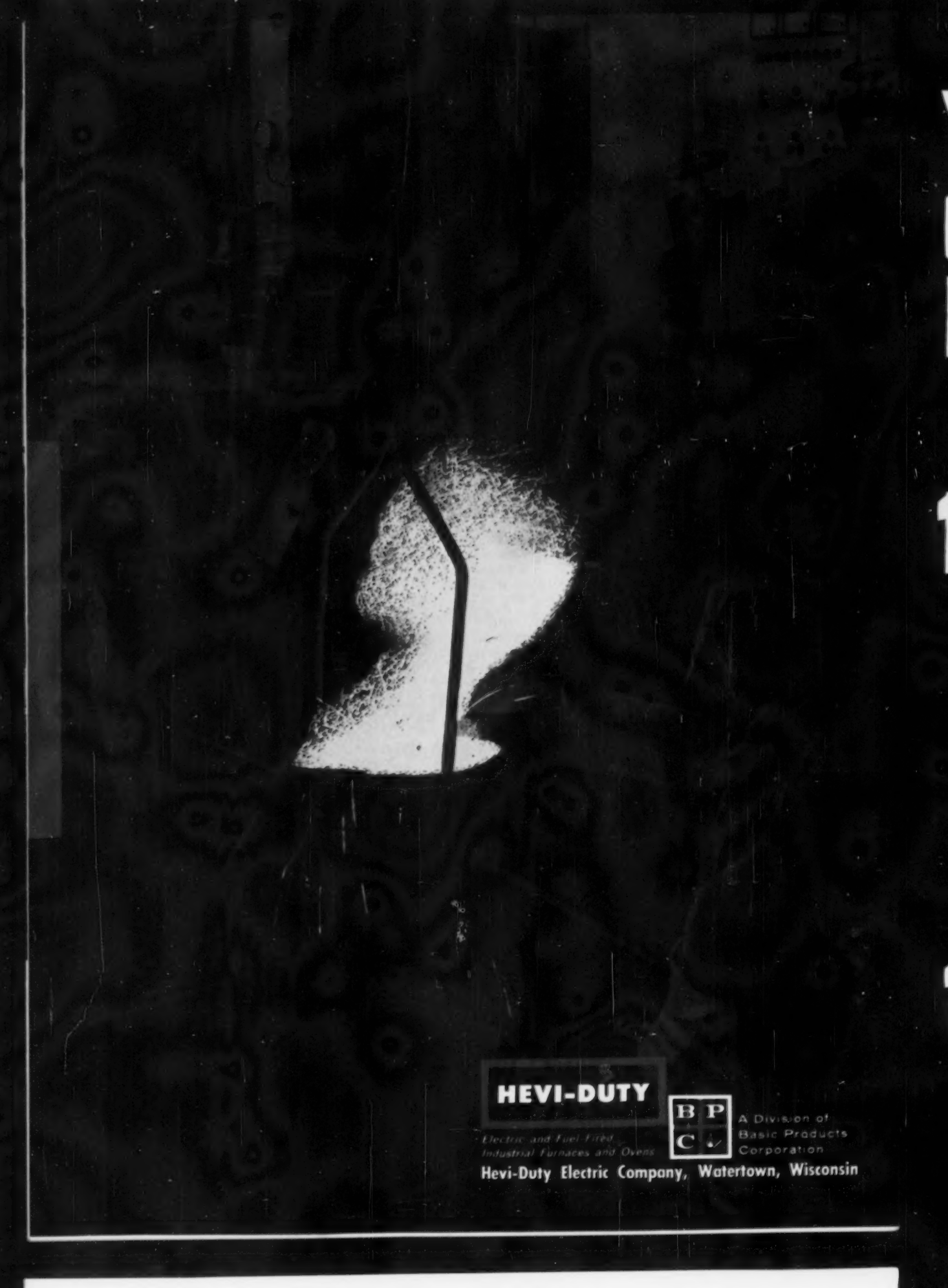
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personals

Julius J. Harwood has been appointed manager of the Metallurgy Dept., of Ford Motor Co. He was formerly head of the Metallurgy Branch, Office of Naval Research in Washington.

H. N. Lander is now assistant director of research at the Youngstown Sheet & Tube Co., Youngstown, Ohio. He previously was with the Graham research laboratory of Jones & Laughlin Steel Corp.

H. P. Leighly, Jr. has joined the faculty of the Missouri School of Mines and Metallurgy as associate professor of Metallurgical Engineering. Dr. Leighly has recently been research metallurgist at the Denver Research Institute and in charge of the instruction of metallurgy at the University of Denver.

Howard O. Beaver has been appointed assistant general superintendent of the Carpenter Steel Co., plant in Reading, Pa. Manager of mill metallurgy at the plant for the past two years, he came to Carpenter in 1948 and was named plant metallurgist in 1951 and production metallurgist in 1957.

Will Mitchell, Jr., has been appointed acting director, research div., of Allis-Chalmers Manufacturing Co. He was previously associate director of the company's research div. Mr. Mitchell has been with the company since 1947.

John E. Schork is now director of manufacturing of Research-Cottrell, Inc. He joined the company in 1956 after prior experience as plant superintendent with Hudson Smelting & Refining Co.

Charles E. Peterson is the new division manager in charge of the Mackintosh-Hemphill div. of E. W. Bliss Co. He joined the company in 1957, and until his present appointment was manager of manufacturing operations.

Anthony Demos has joined Frank Samuel & Co., as manager of metallurgical sales. He was previously with Foote Mineral Co.

Bruce C. Whitmore has joined the staff of the Tonawanda Laboratories of the Linde Co., as a member of the metallurgy group.

J. B. Newkirk of General Electric Co., Schenectady, will assume the duties of professor of chemistry and metallurgical engineering at Cornell University.

A. Nagy and **T. A. Felk** have been appointed to the positions of metallurgical engineers, technical service, at the Bridgeville plant of Universal-Cyclops Steel Corp. Mr. Nagy was formerly research associate, and Mr. Felk was in the metallurgical laboratory.

Edward S. Bunn, chief metallurgist of Revere Copper & Brass Inc., has been appointed general manager of the company's research and development department in Rome, N. Y. He will be responsible for all activities in the department.

Vernon G. Kneeskern is now assistant general manager of Reynolds Metals Co's. reduction div. He will have responsibility for the Massena, N. Y., and Troutdale, Ore., facilities.

M. M. Rand has been appointed manager of carbon electrodes and refractories for National Carbon Co. He was formerly eastern division manager of electrode products.

C. K. Waibel has been appointed assistant manager of the coke plant sales department of the Koppers Co. Inc's., engineering and construction div. He has been a coke plant sales engineer since 1951.

Ralph P. DeVries has been named technical assistant to the manager of quality control at the Watervliet works of Allegheny Ludlum Steel Corp. He was previously supervising process metallurgist, air and induction melting.

Carl E. Wood has retired from the US Bureau of Mines after 35 years of service with the agency. Most of this time has been spent in the Minneapolis area.

Richard R. Moore will receive a Certificate of Merit from the Franklin Institute. Mr. Moore developed a materials fatigue testing machine in 1924 which is said to have revolutionized metallurgical concepts. He is chief metallurgist for the Naval Air Engineering Facility of the Naval Air Material Center at the Philadelphia Naval Base.

William J. Harris, Jr., has been appointed assistant executive secretary of the Division of Engineering and Industrial Research of the National Academy of Sciences-National Research Council. Dr. Harris was formerly executive director of the division's Materials Advisory Board.

John R. Freeman, Jr., vice president metallurgy and research, of the American Brass Co., retired Sept. 30, after more than 30 years with the company. Mr. Freeman will now serve as metallurgical consultant to Anaconda Sales Co., and will represent Anaconda as its technical representative in the Copper Products

(Continued on page 881)

For Early 1961 Publication from McGraw-Hill . . . .

MANUFACTURING PROCESSES

By

James S. Campbell

University of California, Berkeley

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Paul H. Black

Ohio University

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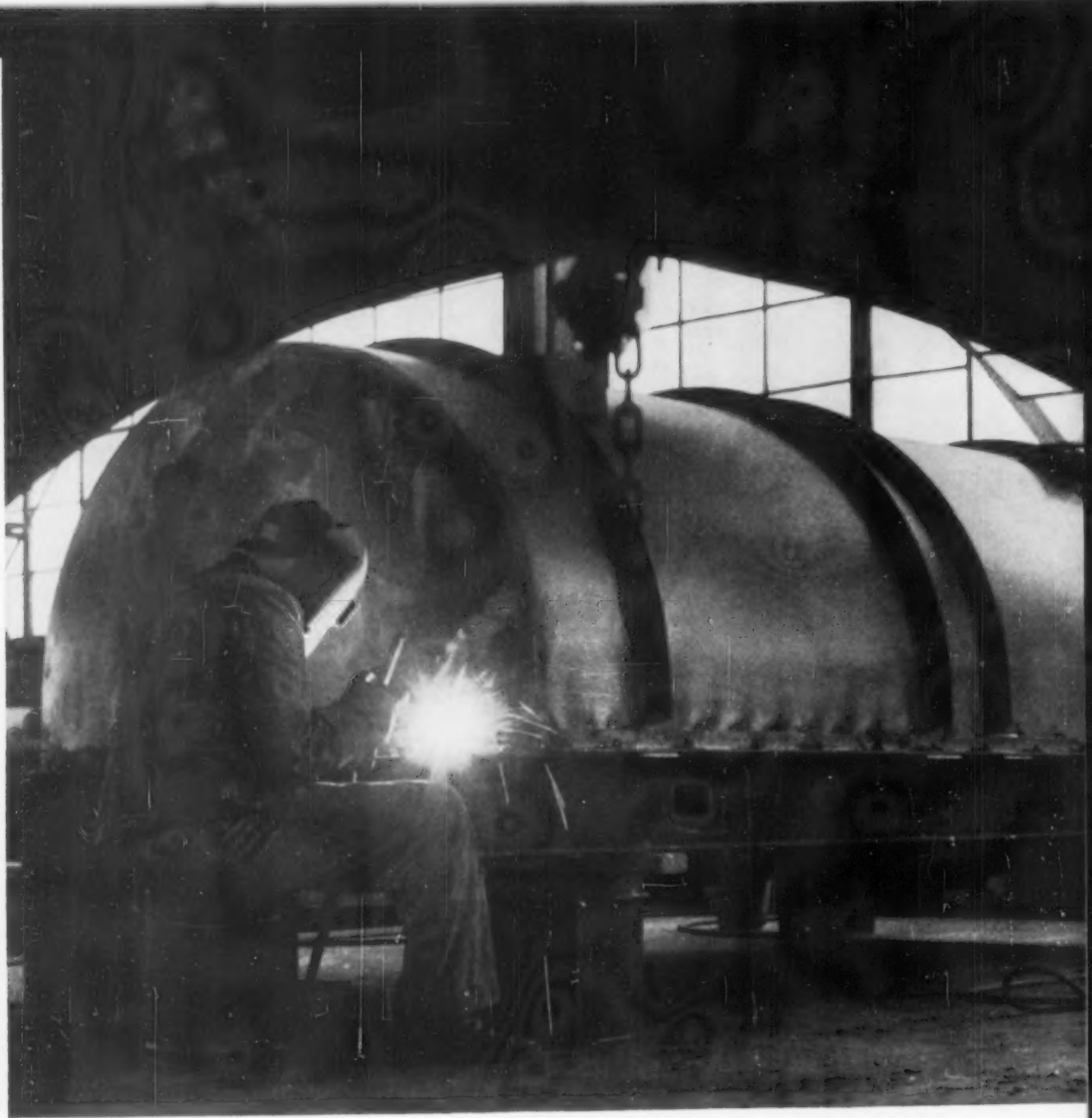

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letters
to the editor

Coulter Counter

In the January 1960 issue of the JOURNAL OF METALS, page 51, the article on Powder Metallurgy appeared and very kindly referred to the Coulter Counter. However the statement that Process Control Services Co., of Elmhurst, Ill., has marketed the Coulter Counter is incorrect, and we would greatly appreciate it if you could possibly retract it, and publish in correction that the Coulter Counter has been marketed by Coulter Industrial Sales Co.

Robert H. Berg
Coulter Industrial Sales Co.

* * *

In-Fab

Your coverage of *In-Fab—For Hot Working Refractory Metals* in the August, 1960 issue of the JOURNAL OF METALS is very much appreciated. However, the article does contain several statements which are incorrect and misleading. Referring to paragraph 2 on p. 636, it should read as follows:

> At present, operations are in the 4000°-4500°F range—the empirically-determined temperatures for hot working molybdenum and columbium (85 pct of melting temperature). Further study is expected to bring about the application of higher temperatures—*up to 500°F for tantalum and up to 600°F for tungsten.* The protective argon atmosphere is effective in deterring both the oxidation and the absorption of gaseous impurities at these elevated temperatures.

You will note that the major problems in this paragraph are the indications that the metals involved will be hot worked at temperatures above their respective melting points. In reference to cobalt, it melts at 3430°F and obviously cannot be worked in the 4000°-4500°F range. As a matter of fact, there is no intent at the present time to process any cobalt base materials. The room has been specifically built for the refractory metals.

In paragraph 3, reference is made to the size of the enclosure. Actually, the size is 42 ft wide, 97 ft long, and *23 ft. high.* Apparently the twenty-

(*Continued on page 883*)

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editorial

A SUCCESS STORY

or

How a Committee Should Function

Technical sessions on semiconductors have been held at AIME meetings almost every year since 1954. Initially these were planned by members of the Metal Physics Committee of IMD. But in May 1957, five members of that committee set about forming a separate Semiconductors Committee to provide for an exchange of technical information through sessions, conferences, and publications.

Organized under the chairmanship of W. C. Hittinger in February 1958, the committee's first endeavor was a symposium on *Government-Sponsored Research in Semiconductor Metallurgy*, followed by a luncheon and round-table discussion, at the 1958 Fall Meeting. Attendance was less than 100, but interest was high. Thus, the new committee felt that a separate conference on semiconductor metallurgy with a published volume of proceedings was desirable.

With the co-sponsorship of the Boston section of AIME and with working capital borrowed from the IMD fund, they staged a technical conference on *The Properties of Elemental and Compound Semiconductors* in Boston late in August 1959. It consisted of four half-day sessions, a panel discussion, and a well-organized visit to Lincoln Laboratories. Registered attendance was almost 500. Proceedings of the conference was published as Volume Five of The Metallurgical Society Conference Series. Copies were mailed to all registrants, and the volume enjoys a good outside sale. Registration fees were sufficient to return all borrowed funds, pay expenses, and retain a comfortable balance.

Following this conference, the Semiconductors Committee was enlarged to 19 members, thereby providing a coast-to-coast geographical distribution. New officers were selected, and planning was started for a second conference to be held in 1960. The meeting, again held in late August in Boston was devoted to *The Metallurgy of Elemental and Compound Semiconductors*. Details of this second successful meeting are given on p. 873 of this issue of JOURNAL OF METALS.

It is the intent of the committee to continue these annual conferences and vary their location so that all segments of the industry will be served. Thus, the 1961 conference will be held in Los Angeles from August 30–September 1 with the Southern California section of AIME acting at host. Tentative locations for subsequent years are Philadelphia in 1962, Chicago in 1963, and Boston in 1964.

The committee also expects to arrange technical sessions of contributed papers at regular AIME meetings. And it is representing The Metallurgical Society in the organization of a symposium on *Thermoelectric Energy Conversion*, to be co-sponsored by several professional societies and the Department of Defense in Dallas on January 8–12, 1961.

The first two conferences have put the finances of the Committee on a firm basis. Organization has been quite informal, and it is felt best to avoid over-formalization; however, consideration is being given to the adoption of a simple set of bylaws.

D. C. Jillson, Chairman
IMD Semiconductors Committee

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TRANSACTIONS

OF THE METALLURGICAL SOCIETY OF AIME

Recently Accepted TRANSACTIONS Papers

The Isothermal Transformations of Ti-2.5 Al-16V and Ti-4 Al-3 Mo-1V, by L. E. Tanner (IMD)

Microstructure of Unidirectionally Solidified Al-$CuAl_2$ Eutectic, by R. W. Kraft and D. L. Albright (IMD)

Copper, Nickel and Iron Alloys for the Quantitative Recovery of the Platinum Metals in Ores and Concentrates, by M. E. V. Plummer, J. M. Kavanagh, J. C. Hole and F. E. Beamish (EMD)

Reaction of Pure Tantalum with Air, Nitrogen, and Oxygen, by W. M. Albrecht, W. D. Klopp, G. B. Koehl, and R. I. Jaffee (EMD)

The Effect of Plastic Deformation on the Electrical Resistivity of Composite Silver-Alumina Alloys, by H. R. Peiffer (IMD)

Effect of Grain Size on the Deformation of Polycrystalline Silver Chloride at Various Temperatures, by R. D. Carnahan, T. L. Johnston, R. J. Stokes, and C. H. Li (IMD)

Reduction Kinetics of Magnetite in H_2H_2O-N_2 Mixtures, by W. M. McKewan (ISD)

Catastrophic Oxidation of Stainless Steel in the Presence of Lead Oxide, by John C. Sawyer (IMD)

Uranium Solubility in Bismuth-Base Liquid Solutions, by A. F. Weinberg, R. J. Van Thyne, and R. E. Steiner (EMD)

Effect of Quenching on the Grain-Boundary Relaxation in Solid Solutions, by A. S. Nowick and C. Y. Li (IMD)

Cube Texture in Ultra-Thin Molybdenum Permalloy Tape, by P. K. Koh (IMD)

Dispersion Strengthening of Copper by Internal Oxidation, by Oliver Preston and Nicholas J. Grant (IMD)

Redetermination of the Chromium and Nickel Solvuses in the Chromium-Nickel System, by C. J. Bechtoldt and H. C. Vacher (IMD)

Oxidation of Niobium (Columbium) in the Temperature Range 500-1200°C, by Per Kofstad and Hallstein Kjöllesdal (IMD)

The Rates of Formation and Structure of Oxide Films Formed on a Single Crystal of Iron, by J. Bruce Wagner, Jr., Kenneth R. Lawless, and Allan T. Gwathmey (IMD)

An Evaluation of Procedures in Quantitative Metallography for Volume-Fraction Analysis, by John E. Hilliard and John W. Cahn (IMD)

The Structure of Bainite in Hypoeutectoid Steel, by S. J. Matas and R. F. Hehemann (IMD)

A Study on the Texture Formation in Rolled and Annealed Crystals of Silicon-Iron, by Hsun Hu (IMD)

Liquid-Solid Phase Distribution Studies in the System Iron-Lead, Cobalt-Lead, Chromium-Tin, and Nickel-Silver, by David A. Stevenson and John Wulff (IMD-EMD)

Phase Relations in the Titanium-Aluminum System, by Elmars Ence and Harold Margolin (IMD)

Effect of Copper on the Corrosion of High-Purity Aluminum in Hydrochloric Acid, M. Metzger, G. R. Ramgopal, and O. P. Arora (IMD)

The Free Energy of Formation of CdSb, by Richard J. Borg (EMD)

Easy Glide and Grain Boundary Effects in Polycrystalline Aluminum, by Robert L. Fleischer and William F. Hosford, Jr. (IMD)

Effect of Temperature on the Creep of Polycrystalline Aluminum by the Cross-Slip Mechanism, by N. Jaffe and J. E. Dorn (IMD)

Study of the Spectral Emissivities and Melting Temperatures of Osmium and Ruthenium, by R. W. Douglass, and E. F. Adkins (IMD)

Effect of Hydrogen on the Tensile Properties of Iodide Vanadium, by A. L. Eustice and O. N. Carlson (IMD)

The Dependence of Wire Texture in F.C.C. Metals on Stacking Fault Energy, by Norman Brown (IMD)

Effects of Explosive Shock Waves on a Gold-Silver Alloy, by A. S. Appleton, G. E. Dieter, and M. B. Bever (IMD)

Thermodynamic Properties of Cr_3C_2 at High Temperatures, by S. Fujishiro and N. A. Gokcen (ISD)

The Effect of Orientation on the Recrystallization Kinetics of Cold Rolled Single Crystals, by W. R. Hibbard, Jr. and W. R. Tully (IMD)

new books

Books that are marked (•) may be ordered through AIME. Address Irene K. Sharp, AIME Book Dept., 29 W. 39 St., New York 18, N. Y. A discount is given whenever it is possible.

Properties of Elemental and Compound Semiconductors, Edited by Harry C. Gatos, *Interscience Publishers, Inc.*, 340 pp., $8.50, 1960. This volume contains the papers and discussions of a Technical Conference (Sept. 1959) sponsored by the Semiconductors Committee of The Metallurgical Society and the Boston section of AIME. The first four sections of the book deal with metallurgical and chemical aspects; solid state and surface reactions; chemical and lattice defects; and physical properties and device applications. The final section of the book is a record of a panel discussion on the role of dislocations in device properties. •

Flat Rolled Products II: Semi-Finished and Finished, Edited by E. W. Earhart and R. D. Hindson, *Interscience Publishers, Inc.*, 150 pp., $4.00, 1960. This volume contains the papers and discussions of the Second Annual Conference (Jan. 1960) sponsored by the Mechanical Working Committee of The Metallurgical Society and the Chicago section of AIME.

Section one deals with processing to semi-finished products, and includes papers on hot machine scarfing, strip mill ingot and slab size selection, and the scale structures of mild steel strip. Section two deals with processing finished flat rolled products, and includes papers on temper rolling, magnetic materials and unitized automobiles. The appendix contains a speech dealing with retirement opportunities. •

Extractive and Physical Metallurgy of Plutonium and Its Alloys, Edited by W. D. Wilkinson, *Interscience Publishers, Inc.*, 314 pp., $10.50, 1960. Based on the 1959 San Francisco symposium sponsored by committees of The Metallurgical Society of AIME. W. D. Wilkinson of the Argonne National Laboratory contributes a special *Introduction to plutonium metallurgy*, and a 44-page annotated bibliography. Six papers on the extractive metallurgy of plutonium include discussion of conversion of salts and reduction of halides to metal, preparation of metallic plutonium and halides, and removal of fission product elements. Eight papers on the physical metallurgy of plutonium include discussion of alloying behavior, plutonium-cerium and plutonium-zinc phase diagrams, etching, zone refining, and alpha, beta, and gamma plutonium.

Technical Communication, by George C. Harwell, *Macmillan Co.*, 332 pp., $5.00, 1960. Written with the engineering student in mind, this book will also be useful to the practicing engineer and those in some other professions. The first three chapters discuss the qualities of good writing, the organization of material, and the use of exposition. The business letter is covered in detail. There is an extended treatment of reports and information on technical articles, magazine writing, oral communication and the use and preparation of tables and figures. The final section of the book is a manual of general composition, containing a review of the general principles of composition and a glossary of usage. •

The Manufacture of Iron and Steel, Volume Three; Steelworks Fuels, Furnaces, Refractories, and Instruments, by G. Reginald Bashforth, *Chapman & Hall, Ltd.*, London, 246 pp., about $4.95, 1960. This 1959 revision of volume two of the original work necessitated a third volume to accommodate the large amount of new material involved. Thus, volume one deals with iron production, volume two with steelmaking processes, and the new volume three discusses open hearth fuels, liquid fuels, and producer gas; the open hearth furnace, plant and equipment;

(Continued on page 881)

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Revolutionary aluminium process

Publication of British Patent No. 846,189 confirms earlier guesses as to the nature of Aluminium Limited's recently announced aluminum production process, which bypasses the costly intermediate step of converting ore to alumina.

The patent deals predominately with the recovery of the metal from the subhalide gas, and only very broadly with the production of the latter. The patent states, "At appropriate temperature, ordinarily in the range of 1000°C and upwards, and under suitable pressure, which may be atmospheric or sub-atmospheric, the gaseous halide reacts with the aluminium in the (raw) material to produce in gaseous form an aluminium subhalide, e.g. a monohalide.

"The converted gas, which may also contain some unreacted halide, is led to the decomposing region where a suitably lower temperature is maintained, so that the reverse reaction occurs. . . . The metallic aluminium is collected while gaseous normal halide is carried along for recovery and reuse." The patent continues, "It has been found that decomposition of the subhalide . . . can be achieved in a . . . so-called splash-type condenser."

[Readers are referred to a previous Reporter article in the Sept. 1960 issue of JOURNAL OF METALS, p.663.]

French aluminum process

Announcement by the two French aluminum producers, Pechiney and Ugine, indicates that they may beat Aluminium Limited to the punch in the development of a direct ore-to-metal process. Aluminium's pilot plant is scheduled to be completed in about one year, whereas the French producers' plant is almost completed, and they are pushing hard to get the 3000 to 5000 ton-per-year plant into production this year.

Representatives of the French producers say that the Pechiney-Ugine process is covered by patents which have already been granted, indicating that their process may be different from the Canadian process.

Thermoelectric compound

Silver antimony telluride ($AbSbTe_2$), said to be one of the most efficient thermoelectrical materials yet developed, is under study at Bell Telephone Laboratories. The compound is expected to find application in such practical devices as heat-to-power converters, and localized coolers, especially for miniature electronic devices.

The material possesses a very low thermal conductivity; its thermo-electric figure of merit, Z, is reported to be about 1.75×10^{-3}/°C over a range of 200° to 500°C, which is the best yet observed for p-type thermoelements in this range. Silver antimony telluride is described as having a disordered cubic structure exhibiting a thermal conductivity as low as 0.0064 w/cm°C at room temperature, only one-hundredth that of germanium.

New steels

Jones & Laughlin Steel Corp., has developed the *Jalloy-S* series of Mn-Mo constructional steels, which are available in three grades ranging from 90,000 to 110,000 psi yield strength. They are said to combine high strength with an ease of welding and forming. It is expected that they will find application in missiles, ordnance, and industrial equipment. J & L has also introduced the *Jalloy-AR* high-abrasion resistance series.

Westinghouse Electric Corp., has developed the *Kromarc* group of stainless steels, which are claimed to have superweldability and high strength. Westinghouse has made several 1-ton pilot heats of the new compositions. The company expects that the new steels will virtually eliminate the tendency of stainless steels, in such large thick structures as pressure piping and castings, to fissure or crack during their fabrication by welding.

Strategic-Udy plant

New Mylamaque Explorations Ltd., has contracted with Koppers of Canada Ltd. (a subsidiary of Koppers Co. Inc.) and the Strategic Materials Corp. for the construction of a $14-million smelting plant near Kingston, Ont., Canada, for the production of pig iron by the Strategic-Udy direct reduction process. Under terms of the contract, Koppers will handle the technical and operational management of the smelter. Production is expected to begin early in 1962 with an estimated capacity of 150,000 tons of pig iron from ore containing 6 pct TiO_2.

"Stainless" carbon steel

Alphatizing, a new process which gives carbon steel many properties of stainless steel at a cost saving, has been developed by Alloy Surfaces Co. The process is based on a new approach to chromium diffusion, which is said to increase the resistance of carbon steel to heat, corrosion, and wear. The process reportedly supplies an adequate and controllable amount of chromium to the metal surface. The coating becomes an integral part of the metal and produces a true alloy. It is claimed that ductility, weldability, hardness, corrosion, and wear resistance can be developed to the degree desired. Mild or low carbon steels show greatest chromium penetration.

Germanium recovery process

High Purity Metals Inc., has developed an automated process for economically reclaiming high grade germanium scrap. The key to the process is said to be a specially designed solid-gas reactor which insures high quality, intrinsic germanium from which makers of semiconductors can pull single crystals. Four general types of material will be accepted from semiconductor manufacturers for processing: crystal ends, high-grade scrap, broken slices, and high-grade sawdust (single crystal, polycrystalline, or doped germanium forms).

REPORTER

New graphite process

National Carbon Co., is producing a recrystallized graphite which is said to be a promising high-temperature rocket material. Using a new hot-working process the recrystallized graphite is produced with approximately two to three times the high-temperature strength of conventional graphites. The material is claimed to have usable strength up to 5500°F. The company says there are no technological limitations to the size in which recrystallized graphite can be produced.

Armour Research Foundation of the Illinois Institute of Technology has developed a new synthetic binder that reduces the time and cost of making molded graphite. The binder was developed under contract for the Atomic Energy Commission. It is said to cut production time needed to make strong, dense graphite from nearly 90 days to less than 2 hr by eliminating baking, the slowest and most expensive stage in commercial graphite manufacture. Only a small amount of the binder is needed in the new process, and it leaves a virtually ash-free residue.

Curium—242

Martin Co., is building a processing laboratory in Baltimore to produce *Curium-242*, one of the man-made elements which can be used as a source of heat in small auxiliary power generators like the SNAP 1-A and SNAP-3 units built by the company's Nuclear div.

Curium-242 is claimed to have several advantages over *Polonium-210*, the fuel which was used in the first SNAP-3 generator: a longer half life, radiation more effective in generating heat, higher melting point, and lower cost.

Foote research center

Foote Mineral Company is building a $3-million research center at Paoli, Pa. Scheduled for completion in November, the 60,000 sq ft laboratory will have an extensive library and research and development sections for metallography, metal extraction, processing, foundry work, spectrography, metal-working, testing, electrical studies, chemical analysis, electrochemistry, metal organics, and ceramics.

Metal-ceramic composite

Pfaudler Permutit, Inc., has developed a tough metal-ceramic composite which is expected to have broad application in the chemical, nuclear, and aerospace fields.

Called *Nucerite*, the crystalline ceramic is physically and chemically bonded to structural metals, such as cold-rolled steel, Inconel, stainless steel, and molybdenum, tantalum, columbium, and other refractory metals which may require oxidation protection at high temperatures.

The product is said to have unusually high resistance to corrosion, thermo-shock, abrasion, and impact, and reportedly can be applied to tubular goods, flat-rolled items and products of intricate shapes.

Continuous coke furnace

A new technique employing a rotary hearth type furnace, similar to that used in metallurgical operations, has been developed for the continuous production of metallurgical grade coke. A joint development of Salem-Brosius Inc., and New York Mining & Manufacturing Co., the new method is said to require only about a quarter as much auxiliary equipment as other coking processes.

In adapting the rotary hearth principle for coke production, a deep bed of coal is fed onto the hearth, which rotates within a circular shell. Hot exhaust gases from high temperature zones in the furnace are directed to the zone immediately behind the feeding hopper, igniting the coal. Air, directed into the combustion zone through ports in the side of the furnace, supports combustion of the gases in the coal. As the furnace continues to rotate, the coking process progresses. When it is completed, the coke is withdrawn from the furnace and quenched. Coking is completed in one revolution of the furnace hearth.

The two companies will put the process into operation with a pilot-type furnace, and later will build units about 70 ft in diam. Production goal from the 70 ft furnace is about 5000 tons per month of high grade metallurgical coke from about 8000 tons of coal.

New spherical powders

A process for producing spherical powders of metals and metal alloys with particle sizes ranging from 25 to 150 μ and a uniformity of 98 pct has been developed by Linde Co., div. of United Carbide Corp. Starting material for the powders is 1/16 in. diam wire of uniform cross section and free from surface imperfections.

The powders, produced in a totally inert atmosphere, are free from such defects as voids, cavities, and inclusions. Use of the powders is expected to promote: better quality and production rates for sintered parts, reduction in the hazards of handling highly-reactive powders, and improvement in the thrust of solid-fuel rocket propellants through the use of additives such as aluminum in spherical powder form. The use of spherical powders produced under close laboratory controls is expected to serve as a standard to calibrate powder analyzing equipment. Six different powders are already available: copper, aluminum, nickel, 316 stainless, tungsten, and nichrome.

Third Kaldo Plant Near

A new $100-million steel works at Oxelösund—the largest single private investment scheme ever to have been undertaken in Sweden—is rapidly nearing completion. Featured will be two interchangeable 110-ton Kaldo furnaces similar to those recently put into operation at the Sollac plant in France (see pp. 853). This steel plant—owned by the Grängesberg-Oxelösund Co.—is to have a steelmaking capacity of 430,000 tons annually from Kaldo and open hearth furnaces.

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The fantastic accuracies needed by inertial guidance systems for space flight depend on the suppression of gyro drift, the tendency of a gyro to precess from minutely occurring internal torques. Particularly puzzling has been the problem of "jogs," or sudden axial shifts, within gyro spin-axis bearings. Shifts of but one ten-millionth of an inch can cause serious steering error.

Specialists at the GM Research Laboratories have found that the real key to drift lies in the thickness and distribution patterns of bearing lubricating films. Only a tenth of a milligram of oil – equivalent in volume to less than two-thousandths of a drop of water – is required in a gyro bearing, but even this amount unevenly distributed may cause jogs.

Conducting unique studies of single bearings apart from rotor assemblies, GM Researchers use a hydrostatic spindle and special instrumentation to take film-thickness measurements they compare with hydrodynamic theory. Jogs, due to excess oil supply, have been analyzed in relation to surface oil transfer and separator feed control, ball spin orientation, displacement, and differential heating and ball wander.

This experimental and analytical approach is achieving progress toward jog-free, stably distributed, and suitably thick oil films required in high-precision bearings. It is a further example of the critical and advanced research General Motors carries out in seeking "more and better things for more people."

General Motors Research Laboratories

Warren, Michigan

The fluoresced streaks show the disturbed "wake" of the lubricating film during bearing operation. The active part of the film, too thin to fluoresce visibly, averages ten-millionths of an inch in thickness.

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1960 ELECTRIC FURNACE PROGRAM

CONDENSED PROGRAM

(The complete program will appear in the December issue)

Sponsored by the Electric Furnace Committee of the Iron and Steel Division, The Metallurgical Society of The American Institute of Mining, Metallurgical, and Petroleum Engineers

Morrison Hotel, Chicago — November 30 to December 2, 1960

WEDNESDAY, NOVEMBER 30

8:30 am **Registration**

9:30 am **Opening Session**

Welcoming remarks; announcements and reports.

9:45 am **Joint Technical Session— Non-Metallic Inclusions in Steel**

Techniques for the study of nonmetallic inclusions; inclusions in stainless steel; inclusions in high-temperature alloys and ferritic steels; quantitative extraction of non-metallic inclusions from steel; petrographic tools and techniques in examining nonmetallic inclusions in steels.

2:00 pm **Combined Technical Session— Ingot and Special Arc— Factors in Improving Steel Quality; Ferro-Alloy Quality and Standardization Vs. Steel Quality**

Reliability factors in steel usage; ferro-alloy situation as seen by a ferro-alloy producer; hydrogen in alloy additives; non-metallic inclusions in alloy additives; ferro-alloy situation as seen by a steel producer; ferro-alloy quality vs. steel quality; what is being done to standardize types and grades of ferro-alloys?

2:00 pm **Castings Technical Session— Control of Non-Metallics in Steel for Castings**

Treatment of steel with magnesium to ease the sulphur problem; vacuum degassing of 500-lb melts for carbon-steel castings; pressure pouring of steel castings; desulphurizing of steel under production conditions.

THURSDAY, DECEMBER 1

9:30 am **Ingot Technical Session— Some Developments in Steelmaking Processes**

Vacuum degassing for better forgings; a refractory quality control program for steel plant; oxygen as an aid to electric furnace economics; summary of direct reduction processes as applied to electric furnace use; basic brick trials in electric furnace roofs; electric furnace bottoms and their maintenance.

9:30 am **Castings Technical Session— Refining Metal in Air Induction Melting**

Manganese-silicon ratio in air induction melting of stainless steel; slag compositions used in air induction melting; production of low-gas content metal using argon atmosphere; oxidation behavior in high-alloy air induction melting and control with argon blanket; oxygen boil in air induction melting; desulphurization of induction melted plain "C" and low-alloy steels; reduction of C, S, and P through oxidation in air induction melting; optimum desulphurization and dephosphorization in air induction melting.

9:30 am **Special Arc Technical Session Manufacture of Carbide and Phosphorus**

Considerations in the use of large vs. small carbide producing furnaces; operation of a 43,500 KVA carbide furnace at Trostberg, Germany; operation of an electric furnace in manufacturing phosphorus; handling and utilization of phosphorus furnace slag.

2:00 pm **Ingot Technical Session— Stainless Steel**

Cleanliness requirements for 430 stainless steel strip; metallurgical and operating problems in processing stainless 430 strip; controlled oxygen blow in melting of chrome-nickel stainless steels; oxygen practice on stainless steels at Sharon Steel Corp.; fluid mold casting; argon casting for improving steel quality; argon casting at Jessop Steel Co.; exothermic hot topping big-end-down ingots for higher yields.

2:00 pm **Castings Technical Session— New Developments**

Auxiliary meltdown torch in electric furnaces; new developments in the analysis of carbon and sulphur; use of quantovac in rapid analysis; iron ore

(Continued on top of next page)

to steel in the electric furnace by the Madrigal Process; future possibilities in melting.

2:00 pm **Special Arc Technical Session—Manufacture of Ferro-Alloys**

Single stage manufacture of low-carbon ferrochromium silicon; manufacture of Simplex ferrochrome by the vacuum process; manufacturing 75 per cent ferrosilicon in a large submerged-arc furnace; casting procedures for ferro-alloys.

6:30 pm **Reception and Cocktail Party**

7:00 pm **Annual Fellowship Dinner**

Speaker: Charles J. Hunter, General Superintendent, South Works, United States Steel Corp., Chicago, Ill.

FRIDAY, DECEMBER 2

Plant Trip

Chartered buses will leave from the Morrison Hotel at 9:00 am. There is a choice of visiting either the South Works of United States Steel Corporation (limited to 125), or to Lemont Manufacturing Company, Lemont, Ill. (limited to 125).

education

Materials Research Center

One of the nation's first inter-disciplinary centers for basic research in materials has been established at Northwestern University. Known as the Northwestern Materials Research Center (NMRC), it is designed to add to basic knowledge of materials' structure and properties and to educate more PhD's in materials research than are now being graduated in this field.

During its first four years, about half of the organization's operating budget will be paid by the Advanced Research Projects Agency of the Department of Defense. A $3.4 million contract with the Government agency was recently signed.

According to Prof. Morris E. Fine, NMRC chairman, the only research done will be basic in nature, and the center is to be interdisciplinary. Fine explains that design advances in proposed weapons, space, and atomic projects have far outstripped the ability of existing materials to do what is demanded of them. Much more basic research is needed to understand the structure of materials and relate structure to properties. Once formulated, this basic information may be useful in developing better materials for specific jobs.

Concerning NMRC's interdisciplinary nature, Prof. Fine noted that five departments are participating—materials science (formerly called metallurgy), physics, chemistry, electrical and mechanical engineering. Traditionally, in both education and industry, study of materials has been segmented into many channels, such as solid-state physics, metallurgy, and ceramic science. It is hoped that the new *cross-fertilization* at the center will speed research progress.

Materials Conference

A three-day conference on *The Science Underlying the Behavior of Materials* was held at the General Electric Research Laboratories in Schenectady, N. Y. on Sept. 7-9. Thirty-eight educators representing 29 colleges and universities in the U.S. and Canada attended.

Among the subjects discussed at the conference were materials science, materials engineering, high-pressure techniques, fracture of materials, magnetism, new energy conversion systems, and the theory, properties, and application of tunnel diodes. J. R. Townsend of the Department of Defense delivered an address on *The Importance of Materials Science to Defense*.

LITERATURE

The Steel Founders' Society of America has published a new pocket directory of steel foundries in the U.S., Canada, and Mexico. Published biennially by the Society, the volume lists all North American steel foundries, their officers and directors, and the officers, directors, and committees of the Society. Selling for $3 per copy, the directory may be obtained by writing Steel Founders' Society of America, 606 Terminal Tower, Cleveland 13, Ohio.

* * *

A four-page data sheet on new cobalt-base and nickel-base superalloys is available from the Cobalt Information Center. This compilation lists physical and mechanical properties, corrosion resistance, thermal treatment, fabrication, and applications of 13 alloys.

Another of the Center's recent publications is Thermal Shock and Thermal Fatigue of Cobalt Alloys and Cermets. A 65-page compilation of references from 1938 to 1960, it includes a brief introduction, 84 abstracts, 14 tables, and 13 figures.

In the Americas, the literature is available upon request from Cobalt Information Center, c/o Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio. The Centre d'Information du Cobalt, 35 Rue des Colonies, Brussels, Belgium, will honor requests from persons abroad.

Industri Information, the seventh in a series of books, has been published by Aktiebolaget Infinitas, a Swedish publisher. This book is the first part of a collective work which will consist of several volumes concerning ferrosilicon alloys.

The book contains three articles; one on the Norwegian ferrosilicon industry by A. Monrad-AS; an article on ferro-silicon and its applications by E. Feitzer and A. Pelz; and an article on the production of ferrosilicon by H. Hagerup-Jenssen. The entire text is printed in both English and German. Copies of the book, which costs about $1.93, may be obtained from Aktiebolaget Infinitas, P.O. Box 7103, Stockholm 7, Sweden.

* * *

A 32-page bulletin, *Properties of Ti-13V-11Cr-3Al*, has been published by Titanium Metals Corp. of America. The beta-titanium alloy is said to provide burst strength to density ratios exceeding one million. The manual includes data covering the metallurgy, design, and welding and forming characteristics of the alloy. Illustrated with 30 figures and 16 tables, the manual is available on request from Titanium Metals Corp. of America, 233 Broadway, New York 7, N. Y.

* * *

The technical pamphlets listed below have been prepared by Battelle Memorial Institute. They are available through the Office of Technical Services, Department of Commerce, Washington 25, D. C.

DMIC Memorandum 62, *The Effect of Rate of Heating to Aging Temperature on the Tensile Properties of Ti-2.Al-16V Alloy Sheet*. Order OTS PB 161212.

DMIC Memorandum 64, *Recent Developments in Superalloys*. Order OTS PB 161214.

DMIC Memorandum 67, *Development of Refractory Metal Sheet in the United States*. Order OTS PB 161217.

* * *

Russian-English Scientific and Technical Dictionaries: A survey has been prepared for the National

(*Continued on page 840*)

Education

(Continued from page 839)

Science Foundation by A. F. Hubbell of New York University. It offers discussions of types of interlingual dictionaries, the plan of existing Russian-English dictionaries, and recommendations for improvement of future works. Also discussed is the organization of lexicographical projects and a survey of needs in particular fields.

Copies of the 20-page report may be obtained for $0.40 each (US stamps or coin or UNESCO coupons) from Prof. A. F. Hubbell, Gallatin House, New York University, New York 3, N. Y.

The Defense Metals Information Center of Battelle Memorial Institute has prepared the following reports. They are available through the Office of Technical Services, Department of Commerce, Washington 25, D. C.

DMIC Report 46H, *Department of Defense Titanium Sheet-Rolling Program*. This report summarizes the progress made on the program during the period from Jan. 1, 1959 through Dec. 1, 1959. Production of heat-treated alloys has been completed by one producer and is nearing completion by the other two. Three new alloys have been added to the program. They are designed to be weldable alpha alloys with high creep strength. Phase II, the development of design data, has begun. This work is being done on production-aged material by Lockheed Aircraft Corp. The Phase III programs are scheduled for completion in 1960. **Order OTS PB 151087.**

DMIC Report 130, *Selected Short-Time Tensile and Creep Data Obtained Under Conditions of Rapid Heating*. This report is a graphical presentation of selected short-time elevated-temperature strength data obtained under conditions of rapid heating. Stress-versus-temperature curves of tensile strength, yield

(Continued on page 883)

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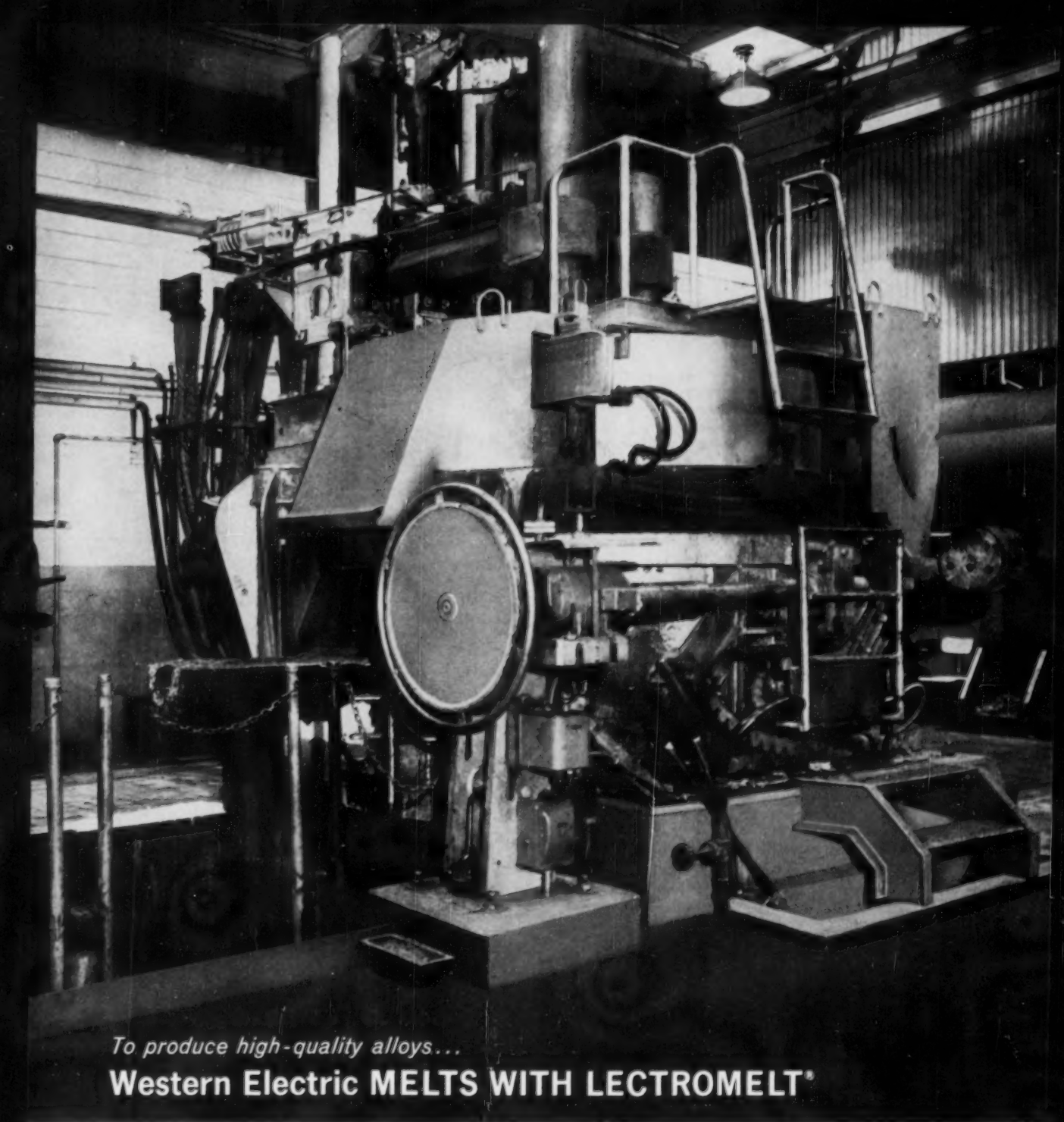

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CONSUMABLE ELECTRODE MELTING OF REACTIVE METALS

Consumable electrode melting of reactive metals was developed because conventional melting methods proved inadequate. Purification of these metals as a function of temperature and pressure is examined, and the problem of the resulting coarse grain size is considered.

by S. J. Noesen

Vacuum arc melting was devised and is being used to purify and consolidate those metallic elements which cannot be processed, in large quantities, in any other way. The most prominent metals in this class are tungsten, molybdenum, tantalum, columbium, zirconium, and titanium. Two properties common to these commercially-important elements, namely their very high melting points and their high degree of reactivity at temperatures near their melting points, make them particularly difficult to consolidate into useful forms.

A quick look at conventional melting methods shows the inadequacies of these techniques. First and foremost, all conventional production techniques have either a ceramic hearth or crucible which have melting points lower than most of the refractory metals. The second, and equally important, objection is the chemical reactivity between these metals and ceramics. Another restriction in melting and consolidating these metals is imposed by their high degree of reactivity with the elements of the atmosphere at high temperatures.

The requirements of a system in which refractory and reactive metals can be melted, refined, consolidated, and cast are as follows: there can be no crucible reaction; there must be a high intensity heat source; and the operation must be carried out in an atmosphere which cannot contaminate the molten metal and preferably one that promotes purification.

The process which was first devised by W. von Bolton, of Germany, in 1903, met all of the above requirements.[1]

He used a water-cooled metallic crucible to contain the molten metal. The instant molten metal came in contact with the crucible it solidified and formed a protective *skull* or shell inside the crucible. Subsequently, and upon provision of proper control, the molten refractory metal was contained in the skull. Therefore no deterioration or interaction with the water-cooled crucible occurred. Because of its high thermal conductivity, copper is now commonly used for the water-cooled crucible. The electrode which von Bolton used was not carbon, but rather was made of the metal to be melted. Very fine droplets of this metal passed through the intense heat of the electric arc and accumulated as a molten pool in the skull.

It is in the arc, where the finely divided droplets are raised to very high temperatures and exposed to vacuum, that some purification occurs. This phase of the purification process is similar to that which takes place during the stream degassing of steel. Von Bolton carried out his melting in a hermetically-sealed glass envelope to which an *air pump* was attached. One can speculate that the pressure of the system during melting was of the order of 1 in. of mercury.

O. A. Simpson further developed the von Bolton furnace and produced approx 1 ton of vacuum arc-melted tantalum which was used in the manufacture of lamp filaments and dental and surgical instruments.[2] In 1909 Weiss and Stimmelmayr, drawing on the work of von Bolton, arc melted tungsten in atmospheres of hydrogen, ammonia, nitrogen, and in a vacuum.[3]

In 1939, W. J. Kroll reported on a technique used to produce titanium ingots.[4] The technique included an inert atmosphere, a consumable electrode, and a water-cooled metallic crucible. R. M. Parke and J. L. Ham, in 1946, reported on the preparation of arc-cast molybdenum by the cold crucible, consumable-electrode technique.[5] They were the first to prepare an arc-cast refractory metal in large quantities under *high* vacuum; i.e., less than 100μ of mercury, using a continuously-formed, consumable electrode. A considerable portion of this historical sketch has been presented by W. E. Kuhn.[6]

Most metallic elements, numerous metalloids, and many intermetallic compounds can be prepared by the consumable-electrode melting technique. Obviously one requirement which is essential in producing a successful consumable-electrode arc melt is that the material to be melted be fabricable into an electrode shape. This electrode must have a low enough resistivity so that the large electrical current necessary to maintain a molten pool in the cold crucible can be carried by the electrode. A partial list of the metals which have been arc cast are: aluminum, silver, copper, iron, nickel, chromium, cobalt, palladium, uranium, titanium, zirconium, thorium, columbium, molybdenum, tantalum, rhenium, and tungsten. In addition, numerous alloys of each of these elements have been produced.

S. J. NOESEN is with General Electric Research Laboratory, Schenectady, N. Y. This paper was presented at the 17th Annual AIME Electric Furnace Conference.

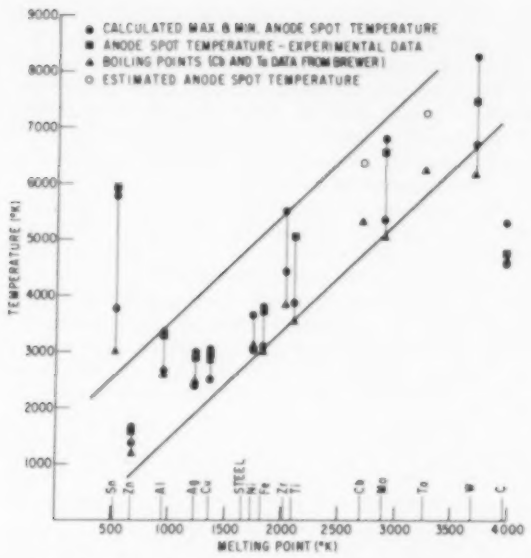
CALCULATED MAX & MIN. ANODE SPOT TEMPERATURE
ANODE SPOT TEMPERATURE - EXPERIMENTAL DATA
BOILING POINTS (Cb AND Ta DATA FROM BREWER)
ESTIMATED ANODE SPOT TEMPERATURE
TEMPERATURE (°K)
MELTING POINT (°K)
STEEL

Fig. 1—Plot of melting points vs. anode spot temperatures.

The metalloids such as boron and silicon can be prepared by arc melting. In this instance, special techniques must be employed since they are dielectric at room temperature. It is only after they have been heated to elevated temperatures that their resistivity drops to a sufficient degree to render them electrical conductors. Intermetallic compounds, even those which have extremely high melting points, can be prepared by this technique. The constituent elements are mechanically fabricated into an electrode.

Purification

Temperature factor

The temperature maintained between the anode and cathode during arc melting is most important in determining the extent of purification, since the three modes of purification are all temperature sensitive. These three modes, plus examples of each are as follows:

1.) vaporization of impurity elements and compounds;

$$Zr + ZrO + W \xrightarrow[\text{temp.}]{\text{high}} W + Zr\uparrow + ZrO\uparrow$$

2.) chemical reactions whose products are gases;

$$MoO_2 + C + Mo \xrightarrow[\text{temp.}]{\text{high}} Mo + CO\uparrow$$

3.) thermally driving off soluble gases.

$$TiH + Ti \xrightarrow[\text{temp.}]{\text{high}} Ti + H_2\uparrow$$

These equations are left unbalanced because equilibrium is never reached since the products of the reactions are continuously removed. The degree to which these phenomena go to completion is directly dependent on temperature and time, and inversely dependent on pressure. In this discussion, only straight polarity—electrode negative—consumable-electrode arc melting is considered. Therefore, the anode temperature (the temperature of the molten bath and its surface) is treated in some detail. For the purpose of analyzing the mechanism by which the final ingot is purified, it is necessary to know the time, temperature, and pressure relationships which exist in and above the molten pool.

Cobine[7] has calculated anode spot temperatures for a number of elements. His calculations were based on high-current short-duration arcs between relatively massive electrodes. Consequently, the specific temperatures which he obtained are probably higher than those encountered in cold-crucible arc melting where a major portion of the heat is extracted by the cooling water. However, his work showed that the anode spot temperature at atmospheric pressure is above the boiling point of each element studied. Therefore, the vaporization rate is high, and very high vapor pressures may occur at the anode spot surface. It should be noted that the entire molten bath surface is not at this temperature, but rather only the centralized anode spot. A plot of Cobine's data is shown in Fig. 1.

Even though these anode spot temperatures are not absolutely correct for cold-crucible arc melting, they do show that the temperature of the molten pool varies from one element to another and is dependent on the vapor pressure of the specific element in question. For instance, the anode spot temperature for tungsten is about twice that of iron. This difference has real meaning to the relative purification which can be achieved in two separate metals. The elements tin and carbon are included in Fig. 1 for general interest. Carbon's anode spot temperature is severely limited because its vapor pressure is extremely high at its melting point. Conversely, tin could have a relatively high anode spot temperature because of its low vapor pressure at its melting point.

It should be noted that the anode spot temperature range shown in Fig. 1 represents that which can be expected at 1 atm. In vacuum arc melting, proportionately lower temperatures can be expected. Until accurate anode spot temperatures and the more important average anode surface temperatures have been determined, the purification process of vacuum arc melting is not amenable to rigorous thermodynamic calculations.

Although the cathode spot temperature is important, its function in straight polarity, consumable-electrode arc melting is limited to controlling the electrode melt-off rate by *gouging* out relatively large molten droplets from the electrode. In the case of the highest melting point elements, thermionic emission is the most important factor governing the current passed by the cathode. Therefore, areas of the electrode tip possessing low thermionic work-functions selectively pass the majority of the arc melting current. In some instances, oxides, nitrides, etc., of elements have lower work-functions than do the parent elements. Consequently, very small, high-current-density cathode spots exhibit positional instability on the cathode surface in seeking out areas of high impurity content, i.e., areas of low-work-function voltage. The net result is a combination of melting and erosion of the electrode tip. This action of the cathode spot has been reported by Johnson[8] and demonstrated by high-speed cinematography by Wood and Beall.[9]

Pressure factor

Since each of the modes of purification which are active in the vacuum arc-melting technique are also pressure sensitive, it is important to know the pressure which exists directly above the molten metal in the crucible. An accurate measurement of this

pressure is not obtained by measuring gas pressures in the furnace body itself or in portions of the vacuum piping system. A pressure measurement taken at a point near the mouth of the vacuum system gives valuable information concerning the gas throughput, i.e. the quantity of gas being removed from the raw material per unit time. However, this measurement is not the same pressure existing at the surface of the molten metal.

If thermodynamic calculations are to be made to ascertain the susceptibility of a given element or alloy to purification, the pool surface pressure must be known. Several investigators have made measurements of this pressure. Gruber,[10] by means of a 0.5-in. inside diam probe which extended down the wall of a 10-in. diam crucible, measured the pressure in the vicinity of the arc while melting a 4 x 4-in. titanium sponge electrode at a rate of 8 lb per min. In this way, it was found that the maximum pressure near the arc was 500μ. Simultaneously, a pressure of 10μ was obtained inside the furnace body. Similarly, Suiter[11] measured pressures with a probe in the vicinity of the arc while melting a 3.2 cm sq, mild steel electrode in a 11.5 cm diam mold. He reported pressures ranging from 15 to 32μ at varying melting rates with accompanying pressures ranging from 13 to 28μ measured at the top of the mold.

Even greater pressure differentials were obtained by Noesen[12] when he used a hollow electrode as a pressure sensing probe while melting molybdenum. In this way the actual pressure, which was imposed on the cathode surface, was sensed. A schematic drawing of the hollow electrode in the melting crucible is shown in Fig. 2. A 2.5-in. diam hydrostatically-pressed and sintered electrode, having a 0.5-in. diam hole along its axis, was melted into a 6-in. diam crucible. At a melting rate of 3.2 lb per min, which resulted in a gas load of 1200 micron-liters per sec, a pressure of 0.8μ was measured above the crucible through the furnace wall. Simultaneously a pressure of 3000μ was measured at the upper end of the electrode-probe. An extension tube attached to the upper end of the electrode-probe passed through a vacuum seal. Various gauges at room temperature were attached to the tube and sensed the 3000μ pressure of the non-condensible gases of the arc aureole.

In this experiment a pressure differential of approx 3000μ was maintained between the arc zone and the furnace body with a pumping load of 1200 micron-liters per sec. The ratio of the cross sectional area of the electrode to the cross sectional area of the crucible was 0.17. The chemical purification which was achieved in the course of this experiment is shown in Table I. Also shown is the calculated quantity of each gas which was removed per unit time, i.e. the gas load of the arc-melting operation in micron-liters per sec. These values were obtained by using the average melting rate of 1460 g per min, the known impurity removal, and assuming that all of the eliminated carbon had reacted with oxygen to form carbon monoxide. The average furnace pressure as measured by a thermocouple vacuum gauge was 0.8μ throughout the melting run. The calculated furnace pressure—0.7μ—was found by referring the above-mentioned calculated gas throughput to a precise pumping capacity curve for the equipment being used. The 3000μ of gas pressure, which was recorded by the gauges on the electrode tube, was assumed to be made up of the non-condensible gases, carbon monoxide, oxygen, and nitrogen.

This experiment exemplifies the necessity of having a small electrode cross section, with respect to the crucible cross section, so that the full effect of the vacuum pumping capacity is utilized in maintaining low pressure in the immediate area of the consumable electrode arc.

The relation between the pressure measured in the furnace body—sometimes even more unrealistically measured at the inlet of the pumps—which is reported as the arc melting pressure, and the actual pressure in the arc zone where purification must take place is dependent on four factors. In order to achieve maximum purification of a given refractory element or alloy, the design and operation of a vacuum arc melting furnace must:

1.) Maximize the annular area between the electrode and the crucible wall, i.e. for a given crucible diameter use the smallest diameter electrode which is consistent with the current carrying capacity necessary to fill the crucible size in question.

2.) Minimize the depth of crucible. A broad shallow crucible is preferred over a long narrow one. Ideally, a bottom pull crucible is used so that, by continuous retraction of the ingot, the arc melting

Table I. Analytical Results of an Arc Pressure Experiment by Noesen[12]

	Electrode Analysis, ppm	Ingot Analysis, ppm	Impurities Removed During Arc Melting, ppm	Throughput Gas Removed,* μ-l/sec
O_2	63	<1	63	470
N_2	2	1	1	15
H_2	<1	<1	<1	<1
C	40	20	20	687 as CO

* Avg melting rate 3.2 lb per min or 1460 g per min.

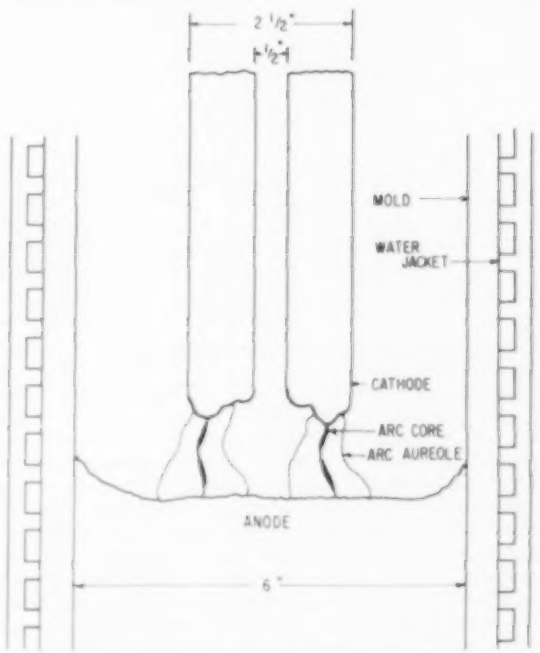

Fig. 2—Schematic drawing of the hollow electrode in the melting crucible.

always occurs in the upper mouth of the crucible.

3.) Minimize the rate of impurity gas additions to the system. Since the raw materials contain a given concentration of impurities which are to be removed, the electrode melt off rate must be minimized.

4.) Minimize the furnace pressure above the crucible. This is accomplished by using short vacuum manifolds with generous diameters and a vacuum pumping system with a high gas throughput.

It is improbable that any two furnaces in existence today are comparable in all four of the above-mentioned characteristics. Therefore, it is of little wonder that there have been conflicting opinions and data concerning pressure-sensitive phenomena in the field of arc melting. The tremendous pressure inhomogeneities, which exist in a single vacuum arc melting furnace, could have been responsible in the past for the conflicting reports concerning the pressures at which diffuse arcs occur.

To demonstrate the importance of controlling the electrode melt-off rate in the purification of a refractory metal, melting experiments involving a molybdenum alloy are cited here. Identical molybdenum electrodes, continuously formed and sintered within the furnace enclosure (equipment and technique described elsewhere),[12] were melted at two different rates by the simple expedient of changing the melting amperage. The electrodes were 1.5 in. in diam, and they were melted into a 6-in. diam crucible giving a very favorable electrode to crucible ratio of 0.06. Table II lists the ingot gas analyses which were obtained when the melting rate was changed. The pressures listed were measured at a point above the mouth of the crucible as before and therefore are only indicative of the actual melting pressures in the arc zone. It can be seen that this pressure increased by two orders of magnitude when the melting rate was increased by a factor of 2.85. These furnace pressures do not increase proportionately with melt rate, i.e. gas evolution, since the vacuum pumping system throughput is not linear with the inlet pressures to the pumps. Undoubtedly, an increased melting rate results in a slightly shorter time for purification. However, it is believed that the poorer purification obtained at the higher rate was due primarily to the much higher pressure of impurities in the arc zone, the magnitude of which is only indicated by the furnace pressure measurements shown in Table II. It is concluded, therefore, that low electrode melt off rates are essential to high purification.

Table II. Molybdenum Ingots Melted at Two Rates

Ingot	Melting Power	Melt Rate	Furnace Pressure	Ingot Analysis, ppm		
				O_2	N_2	H_2
N-76	95kw	1.28 lb/min	4μ	<1	3	<1
Avg of six heats	125kw	3.65 lb/min	392μ	8	6	<1

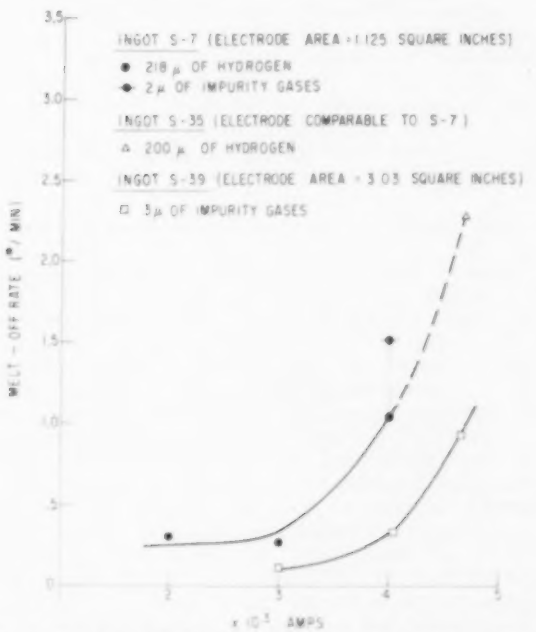
INGOT S-7 (ELECTRODE AREA = 1.125 SQUARE INCHES)
218 μ OF HYDROGEN
2 μ OF IMPURITY GASES
INGOT S-35 (ELECTRODE COMPARABLE TO S-7)
200 μ OF HYDROGEN
INGOT S-39 (ELECTRODE AREA = 3.03 SQUARE INCHES)
5 μ OF IMPURITY GASES
MELT - OFF RATE (" / MIN)
x 10⁻³ AMPS

Fig. 3—Tungsten melt-off rate as a function of current for various electrode diameters and gas additions.

Control of electrode melt off rate

There are five recognized methods of controlling the electrode melt off rate in the consumable electrode arc melting process:

1.) By varying the arc melting power, i.e. voltage, amperage, or both, higher power dissipations yielding higher melt rates;

2.) By varying the electrode diameter, larger diameter electrodes yielding lower melt rates;

3.) By varying the electrode density, higher densities yielding lower melt rates through lowering the electrodes' resistivity;

4.) By the addition of gaseous elements which either change the voltage characteristics of the arc or redistribute the heat dissipation between the anode and cathode surfaces; and

5.) By varying the cross sectional area of the melting crucible, larger diameter crucibles yielding higher melt rates. This last method has been observed very recently and is not completely understood.

The mechanism of the first method is self evident. An example of increased melt rate with increased arc melting power was given in the case of the molybdenum experiments shown in Table II. There is a minimum arc-melting power which is necessary to fill a given-size cold crucible producing a physically sound ingot. This power level determines the amount of heat which the anode spot, i.e. the ingot's molten surface, receives. Arc power in excess of this minimum level adds raw materials, and thus impurities, to the melting crucible at an inordinate rate which is detrimental to final ingot purity, as demonstrated above. In the case of tungsten being deposited into a 4-in. diam crucible, this threshold power level was found to be 140 kw.

The second method of controlling the melt rate has also been demonstrated.[13] It was found that the melt rate, at a given power level while melting tungsten, was appreciably reduced by increasing the cross sectional area of the electrode. The increased mass of the larger electrode is responsible for a lower electrode resistance, thereby reducing its temperature and consequently its melt-off rate. The same phenomenon has been observed in the case of tantalum and molybdenum. This electrode size effect is illustrated in Fig. 3. Under similar conditions of melting power (4000 amp) and furnace pressure,

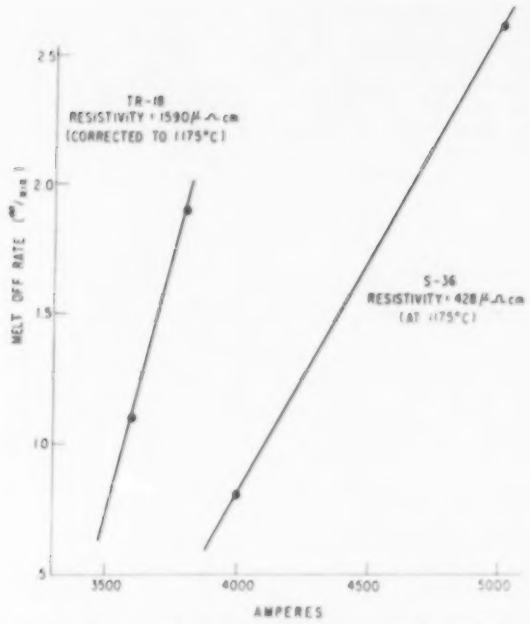

Fig. 4—Electrode melt-off rate as a function of melting current at two electrode resistivities for tantalum.

ingot S-7 whose electrode area was 1.125 sq in. melted at 1.51 lb per min, as compared to 0.33 lb per min for S-39 whose electrode area was 3.03 sq in.

Both electrical and thermal conductivities are increased with increased electrode density. The increase in thermal conductivity probably has only a minor effect on the melt-off rate of the electrode, by conducting slightly more heat away from the molten electrode tip to the furnace component which supports it. On the other hand, increases in electrical resistivity have a strong effect on the electrical energy dissipated within the electrode itself as ohmic losses. Because of the high amperages used in arc melting, slight increases in the electrode resistance cause extremely high temperatures to be generated in the high current flux region adjacent to the small, emitting cathode spots of the electrode. Also heat is generated in the bulk of the electrode itself. Fig. 4 shows the drastic effect of electrode resistivity on melt rate of tantalum. It shows that while melting with 4000 amp, an increase of 3.7 to 1 in resistivity causes the melting rate to go from 0.75 lb per min to approx 3.0 lb per min.

The fourth method of controlling the electrode melt rate is by the addition of small quantities of gaseous elements to the furnace atmosphere. It has been found, in the case of tungsten and tantalum, that the addition of diatomic hydrogen to the melting atmosphere lowers the electrode melt rate at a given arc-melting power. Hydrogen was added to the furnace atmosphere through calibrated leaks and was removed along with the impurity gases by the pumping system. The curve of melt-off rate vs current for ingot S-7, in Fig. 3, shows the effect of hydrogen. With a pressure of 218μ of H_2 in the furnace, the melting rate of 4000 amp was 1.04 lb per min. However, when the hydrogen supply was discontinued and the furnace pressure was reduced to 2μ, because of impurity gases alone, the melt-off rate increased to 1.51 lb per min. Ingot S-35 was melted using an electrode comparable to that of S-7. The melting was done in 200μ of H_2, and its combination of amperage and melt-off rate forms a realistic extension of the S-7 hydrogen curve.

The lower melt-off rates, because of the presence of hydrogen, are attributed to the cooling effect which the dissociation of hydrogen has on the extremely hot cathode spots of the electrode tip. The dissociation of hydrogen is highly endothermic, requiring 102,700 cal per mole. Empirical observations lead to the conclusion that at least a portion of the monatomic hydrogen thus produced recombines at the cooler anode spot (ingot molten surface), giving up some heat of association. This shift in the relative heat distribution from cathode to anode is extremely desirable if a low melting rate is preferred. An additional advantage in having hydrogen present, especially the very active monatomic form, is its strong reducing potential. The reduction of oxygen is especially important in the case of tungsten and molybdenum.

The final known method of controlling the electrode melt-off rate is by varying the diameter of the crucible. Recently it was observed that a 92-pct dense tungsten electrode, whose cross sectional area was 1.03 in. sq, did not show any perceptible melt-off rate while passing 5500 amp at 44 v through it. This observation was anomalous, since past experience dictated that at the power level used, an electrode melt rate of approx 2 lb per min should have been obtained. Upon analyzing all of the conditions existing during previous melting experiments, it was found that only 4-in. diam crucibles had been used to melt tungsten, and in the present melting attempt a 2.25-in. diam crucible was being used. As a result, several melting experiments were initiated, and it was found that crucible diameter did have a strong effect on the melt-off rate of tungsten electrodes. The electrode which exhibited no melt rate in the 2.25-in. crucible was melted, as previously predicted, in a crucible 3.75 in. in diam. Fig. 5 shows that a melt rate of 1.93 lb per min was obtained with the very same electrode that seemed to be completely refractory in the 2.25-in. crucible. In attempting to melt the 92-pct dense tungsten electrode into the smaller crucible, the current was increased to 5500 amp. Consequently, the crucible was damaged by the excessively high currents employed. Next the electrode density was varied. By using a 65-pct dense tungsten electrode, the smaller crucible was successfully filled using 4600 amp at 41 v.

The reasons for the crucible size effect are not at all clear at this time. Undoubtedly, the larger thermally radiating pool surface of the larger crucible is a factor. However, it is believed that a more important factor is a possible redistribution between the fraction of current which passes as thermionically emitted electrons from the cathodes and the fraction which passes as positive ions impinging on the cathode. With larger molten pools, more surface is available from which metallic atoms can evaporate. Some of the atoms which are liberated from the anode surface will be thermally ionized and a certain fraction of the neutral ones will become ioized by direct electron impingement. Those atoms which are ionized will fall through the potential gradient of the arc toward the cathode. Upon impingement with the electrode, the ions will give up their kinetic and thermal energy. The added energy received by the electrode will raise its temperature and increase its melt-off rate.

Vaporization of impurity elements and compounds

The vaporization of impurity compounds represents another mode of purification operating during vacuum arc melting. This phenomenon takes place when the vapor pressure of the impurity compound is greater than that of the base metal at the temperature of the anode surface. Several oxide-metal combinations in which the ratio of oxide vapor pressure to base metal vapor pressure is greater than one at approx 1800°C are as follows:

$$\frac{CbO}{Cb} = 10, \frac{TaO}{Ta} = 10^{c}, \frac{WO}{W} = 10^{2}, \frac{ZrO}{Zr} = 10^{2} \quad [4]$$

Conversely, if the ratio of the vapor pressure of the metal to that of its oxide is greater than 1, as in the case of $\frac{Zr}{ZrO_2} = 10$ at 2000°K,[5] no purification can be expected. In each case the vapor pressure of the impurity, whether an element or a compound, must be determined at the probable anode surface temperature of the base metal in question. In this way, it is possible to determine the purification which can occur.

When an impurity compound is vaporized from the molten metal surface, it is free to condense on any of the three surfaces: the inside of the furnace body, the advancing consumable electrode, and the cold walls of the crucible above the position of the molten metal. Of the three, because of surface temperature and geometrical considerations, the crucible wall presents the most convenient surface on which the impurity compounds can condense. A definite concentration of impurity compounds on ingot surfaces as a result of condensation on the inner crucible wall has been observed. An example of this is shown in Table III.

In all tantalum melting in which the raw material contained large quantities of oxygen and carbon, the resulting ingot had an extremely hard, loosely adhering skull whose analysis showed excessive quantities of both oxygen and carbon. It is presumed that the impurities existed in the raw material as the oxide and carbide of tantalum, and that these compounds were transferred to and thus concentrated at, the crucible wall by the process of vaporization and condensation. Fig. 6 shows two unalloyed tantalum ingots. One, marked TR-13, exhibits the concentrating effect just mentioned. The dark, lacy material on the ingot surface contains 14,500 ppm O_2. The second ingot marked S-36 was made from particularly pure raw materials and shows practically no hard surface. The hard surface skull can be removed from the ingot surface by mechanical means leaving a purified ingot, whose composition is consistent with the time, temperature, pressure relationship existing in the furnace at the time of the melt.

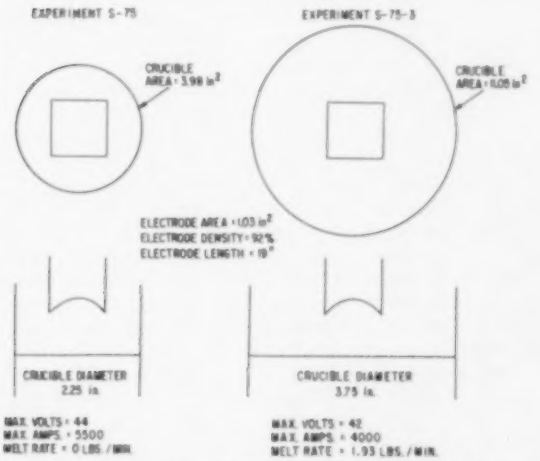
EXPERIMENT S-75
CRUCIBLE AREA = 3.98 in²
EXPERIMENT S-75-3
CRUCIBLE AREA = 8.09 in²
ELECTRODE AREA = 1.03 in²
ELECTRODE DENSITY = 92%
ELECTRODE LENGTH = 19"
CRUCIBLE DIAMETER 2.25 in.
CRUCIBLE DIAMETER 3.75 in.
MAX. VOLTS = 44
MAX. AMPS. = 5500
MELT RATE = 0 LBS./MIN.
MAX. VOLTS = 42
MAX. AMPS. = 4000
MELT RATE = 1.93 LBS./MIN.

Fig. 5—The effect of crucible diameter on tungsten melt-off rate.

Results of arc melting

It has been demonstrated that the temperature of melting (temperature of the anode spot) varies with the base metal being melted. In addition, it has been shown that the degree of purification attainable is inversely proportional to the crucible pressure as indicated by furnace body pressure. Therefore, two generalizations concerning purification during vacuum arc melting can be made:

1) The lower the vapor pressure characteristics of a given element, the higher will be the anode spot temperature. Therefore, because it has the lowest vapor pressure characteristic, tungsten's anode spot temperature is very high, making it particularly susceptible to purification by vaporization, and/or chemical reaction of impurities contained in it.

2) High furnace gas pressure, due to one or a combination of three factors—high impurity level in the raw material, high melting rate, or low vacuum pumping capacity—impedes the removal of impurities normally removed by the process.

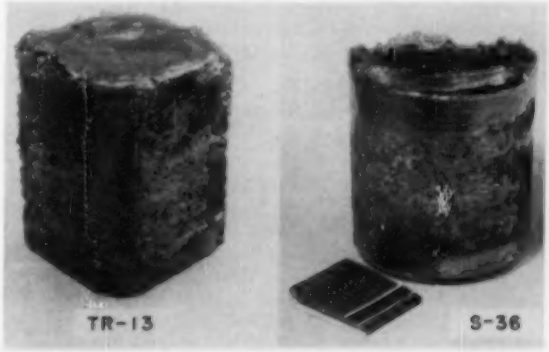

Fig. 6—Tantalum ingots showing difference in impurity skull.

Table III. Concentration of Impurities in the Skulls of Tantalum Ingots in ppm

	Raw Material				Ingot Skull				Ingot			
Ingot	O_2	N_2	H_2	C	O_2	N_2	H_2	C	O_2	N_2	H_2	C
TR-13	1030	33	300		14,500	148	<1		372	18	2	20
TR-16	3360	400	190	250	14,100	560	28	520	1850	170	3	20
TR-20	2000	50	32		9,330	220	<1		740	22	1	

Fig. 7—Fractograph of oxide-coated molybdenum grain boundary surface.

Fig. 8—Pure molybdenum ingot (N-48) 5½ in. diam x 5 in. high, before direct forging.

Typical melting results for a number of metals, including two non-refractory metals, are shown in Table IV.

The cold-crucible arc-melting technique produces ingots which have very coarse, columnar grain structures. Usually the coarser structures are associated with the higher melting point elements. So far it has not been possible to obtain, in ingot form, the very fine grain attainable in powder compacts. Work is under way to induce grain refinement in arc-cast refractory elements by the application of vibration during arc casting. But, as yet, positive results have not been obtained in the case of the high-melting-point refractory metals. Significant grain refinement has been accomplished in the case of steels using vibration.[16] In large molybdenum ingots, grains 0.25 in. to 0.5 in. in diam and 2 in. to 4 in. long are normally encountered.

With such large grains and thus relatively small grain surface per unit volume, the impurities which are present upon solidification and which have low solubilities in the solid base metal, become highly concentrated at grain boundaries. These intergranular constituents may induce intergranular brittleness. Elements, such as molybdenum and tungsten, whose oxygen solid solubility is severely limited, suffer from intergranular brittleness owing to impurity concentration at grain boundaries. Coarse grained molybdenum shows extreme intergranular brittleness when the oxygen content is above 10 ppm.

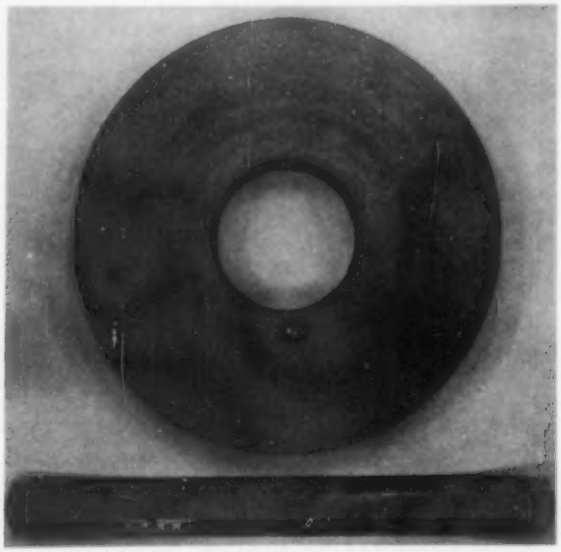

Fig. 9—Pure molybdenum (N-48) after direct forging and machining into final shape.

Fig. 7 shows a grain surface at a magnification of 2000X which is heavily coated with an embrittling oxide phase. This surface was obtained by fracturing an as-cast molybdenum specimen.[17] The resulting fractures predominantly followed grain boundaries. The grain surface which is shown in Fig. 7 contains much more second phase than is necessary to make molybdenum brittle. The oxide phase is in the form of lenticular-shaped particles on the grain surface. The oxygen content of the gross specimen (by vacuum fusion analysis) was 254 ppm.

The tri-axial compressional loading inherent in the extrusion process makes extrusion the preferred process by which the as-cast structure of many arc-melted refractory alloys can be broken down. Once the as-cast structure is destroyed with an accompanying dilution of impurities at the grain boundaries, as a result of increased grain boundary area, more conventional fabrication techniques can be used to obtain the final physical configuration desired.

When the grain boundary contaminants are reduced to very low levels, such as less than 1 ppm of oxygen in the case of molybdenum, direct forging of the as-cast ingot can be accomplished. Figs. 8 and 9 show a 5.5 in. diam by 5 in. high ingot before and after direct forging. The ingot was heated to 1475°C in hydrogen, placed on the forging anvil in air, and upset into a flat disc 12 in. diam by 1.125 in. thick in seven heats.

Conversely, some refractory elements, such as tantalum and columbium, have a greater ability to hold oxygen in solid solution. Therefore, grain boundary concentration of oxygen, due to oxygen rejection into the remaining liquid ahead of the advancing liquid-solid interface does not occur during solidification, even when relatively high amounts of

Table IV. Purification Achieved in a Single Pass Through a Vacuum Arc Melting Furnace*

Element, Heat	Approx Anode Spot Temp	Melting Rate, lb/min	Furnace Pressure, μ	Gas Volume, μ-l/sec	O_2, ppm	N_2, ppm	H_2, ppm	C, Pct	
Cu	2750	2.7	1.3	161.2	1	15	<1		Electrode
(S-22)					1	2	<1		Ingot
CrMoNi Steel	3350**	2.63	1.2	719.12	50	27	1	0.300	Electrode
(S-5)					14	13	<1	0.320	Ingot
Cb	4050**	1.28	2.5	60.3	104	38	<1	0.004	Electrode
(S-29)					98	33	1	0.006	Ingot
Mo	6000	3.18	1.3	687	63	2	<1	0.004	Electrode
(S-2)					<1	1	<1	0.002	Ingot
Ta	6500**	1.63	4.4	21,314	2000	50	32	190	Electrode
(TR-27)					117	17	<1	10	Ingot
W	7500	1.98	1.0	941	9	3	<1	0.005	Electrode
(S-7)					<1	<1	<1	0.001	Ingot

* Furnace located at the GE Research Laboratory.
** Estimated from the plot in Fig. 1.

oxygen are present. Tantalum has shown no indication of intergranular brittleness even when the oxygen content was as high as 500 ppm. The oxygen, for the most part, stays in solution and results in solid solution hardening. A plot of oxygen content vs hardness for tantalum is shown in Fig. 10.

Summary and conclusions

Most metallic elements can be vacuum arc melted. A very few elements, those with low melting points and relatively low vapor pressures, are not adaptable to consumable-electrode melting. These elements, however, can be melted using the inert electrode technique. Vacuum arc melting is an ideal method of producing intermetallic compounds. In addition, metalloids such as boron and silicon have been arc cast. However, special preheating techniques must be employed prior to arc melting to impart some electrical conductivity.

The degree of purification which can be achieved by this process is dependent, through thermodynamic and kinetic considerations, on the time, temperature, and pressure relationships which exist in the arc zone and the molten pool during melting. The temperature which exists is in turn dependent primarily on the characteristics of the base metal making up the consumable electrode. Therefore, a given impurity element or compound can be removed more completely from high arc temperature tungsten than from iron whose arc temperature is lower.

At the present time, arc melting is the only commercially feasible method of producing large quantities (ingots weighing 8000 lb are regularly produced) of high-purity refractory metals and their alloys. But electron beam melting is potentially superior to vacuum arc melting as is zone melting, with respect to ultimate purity obtainable. This is due to the fact that the time of exposure to vacuum and temperature can be independently controlled in the case of electron beam melting.

The extremely coarse grain size resulting from vacuum arc melting of refractory metals represents the most serious shortcoming of the process. Coarse grains are detrimental to the mechanical properties of the cast metal. Grain refinement can be obtained by deformation and recrystallization of the cast product; however, the very fine grain size characteristics of powder metallurgy products has not been achieved. Effort in the area of grain refinement of as-cast refractory metals could be very rewarding since the need is great.

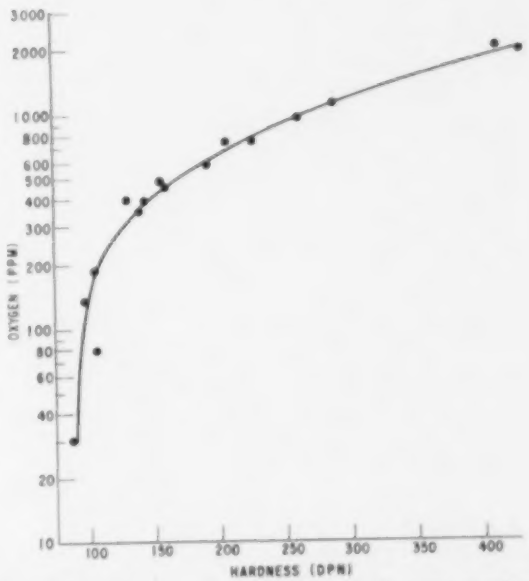

Fig. 10—Plot of oxygen concentration vs. hardness for tantalum.

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A Prepared Discussion

on "Consumable Electrode Melting of Reactive Metals," A Paper by S. J. Noesen

by E. W. Johnson

A rebuttal by Mr. Noesen will be published in next month's JOURNAL OF METALS.

Mr. Noesen's accomplishments in the area of melting and casting the highly refractory metals are in the same tradition and of the same stature, I feel, as the historic contributions made by von Bolton, Kroll, and Parke and Ham. Mr. Noesen has recently demonstrated the melting and casting of 4-in. diam ingots of tungsten, the most refractory of all metals, by a vacuum arc melting process that is very simple and at the same time dependable and effective. This process will undoubtedly be used for producing a wide variety of refractory metals and alloys.

In this discussion I will try to relate some of Mr. Noesen's results on refractory metals to the vacuum arc melting of steel. This is possible in spite of the differences between these metals, because there is a great deal of common ground in the area of mechanisms of arc behavior and metal purification.

Thermal factor

Mr. Noesen has pointed out that metal purification during vacuum arc melting depends on two parameters which we may call *thermal* and *vacuum*. Thermal purification results from raising the metal temperature, or superheating. Mr. Noesen has placed considerable emphasis on the concept that the arc *anode spot* temperature is determined by the metal vapor pressure characteristics, or by the vaporization energy. Apparently, this concept is based on an assumed arc mechanism in which the anode spot area is extremely small and heated very intensely, such that its temperature is limited ultimately by the loss of energy (and material) due to metal vaporization.* Mr. Noesen's Fig. 1 indicates that the resulting anode spot temperature should be approximately double the melting point on an absolute temperature scale.

The concept of an extremely small and intensely heated anode spot is valid when the arc is at atmospheric pressure. In vacuum, however, the arc anode area is no longer strongly constricted but is spread out over a very broad area of the anodic electrode. This larger area is heated quite uniformly to only a moderate temperature. Under vacuum arc melting conditions, therefore, the anode temperature will practically never be high enough to be limited by metal vaporization.

The above contrast between the atmospheric and vacuum behavior of the arc is seen in the accompanying Figs. 1 and 2, which were reproduced from color movie films of arcs. Fig. 1 shows an arc between horizontal, 0.625-in. diam steel electrodes in argon at 1 atm pressure—the cathode is at the left and the anode at the right. Both electrodes are seen to emit sharp, highly luminous flames from small areas or *spots* on the respective electrode surfaces. These flames are presumably constituted of metal vapor streaming rapidly from the respective spots. At 1 atm pressure, therefore, it is quite reasonable to conclude that the temperatures of both the anode and the cathode spots are limited by metal vaporization, as Mr. Noesen has suggested.

The arc in Fig. 2 is at 1600 amp between a 2-in. diam vertical steel cathode and a 9-in. diam anodic crucible. The vacuum pressure of CO gas was 0.1 mm Hg.

The marked difference in appearance between the arcs in Fig. 1 and 2 is due primarily to the difference in the gas atmosphere pressure. In the case of the vacuum arc in Fig. 2, the electrode end is enveloped by a diffuse cathode flame that is far less

* Such a mechanism is specified in the Cobine paper (Noesen's ref. 6), on which Mr. Noesen's computations are based. Mr. Cobine specifically excludes from his treatment low-pressure diffuse arcs of the kind here considered.

E. W. JOHNSON is with the Westinghouse Research Laboratories, Pittsburgh, Pa. This prepared discussion was presented at the 17th Annual AIME Electric Furnace Conference.

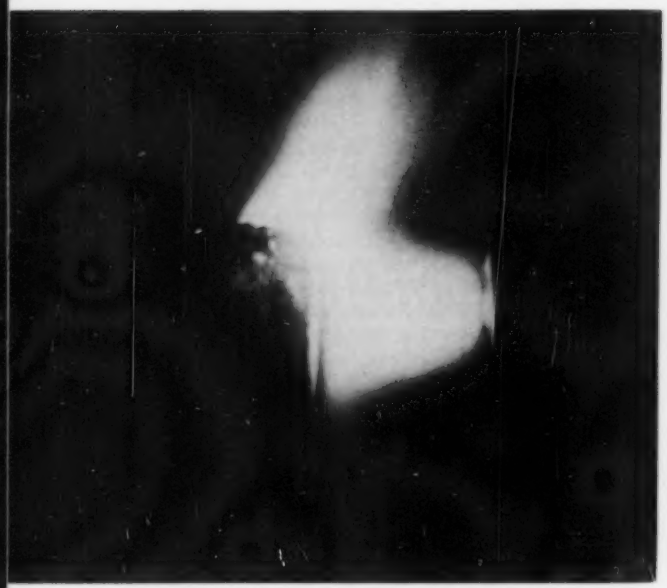
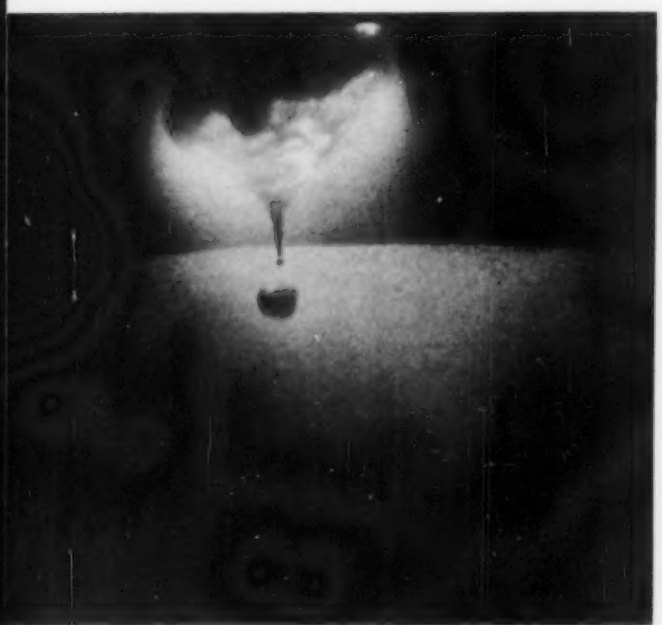


Fig. 1—(Above) 180 amp arc between ⅜ in. diam horizontal steel electrodes in 1 atm argon. Cathode at left.

Fig. 2—(Below) 1600 amp vacuum arc between 2 in. diam cathodic steel electrode and upright sheet-steel anode.



sharp and less luminous than the cathode flame of the atmospheric pressure arc in Fig. 1. Moreover, the anode flame of the vacuum arc, if any, is so weak as to be undiscernible.

The broadness of the vacuum arc anode terminal is easily seen in Fig. 2 from the size of the bright area of the container wall just below (and behind) the electrode tip. At the large arc gap employed in this experiment, the anode terminal did not remain on the pool surface but moved up onto the container wall. This *positional instability* phenomenon has been previously described[1]. The result is that the pool freezes and the anode heat is directed entirely to the wall. The wall in Fig. 2 was merely a 0.050-in.-thick steel sheet which was standing freely without any water cooling. The glowing area was heated to nearly the melting point of the steel and in fact the upper edge of the sheet is just melting. Thus, the anode temperature was just at the melting point, not at double the melting point.

In a vacuum arc furnace, the anode heat is similarly spread over a sizeable area of the pool instead of concentrated at a single tiny point. Thus, the degree of superheat of the pool is probably modest at best, being no higher than that obtained in more conventional melting processes such as induction melting and the open hearth.

The plot in Mr. Noesen's Fig. 1 has been used to explain the fact that the pool temperature during the melting of tungsten is approximately double that obtained with iron. The actual reason for this is to be found in the respective power levels employed. The arc power commonly used for melting tungsten is about three times that used for producing the same size ingots of steel, and the higher pool temperature would naturally be expected on this basis alone.

It was suggested by Mr. Noesen that the molten metal leaves the electrode as "very fine droplets" that are highly superheated and thus thermally purified as they fall through the arc. Our observations have indicated, however, that nearly all the metal falling from the electrode is in the form of rather large drops. A typical example is seen in Fig. 2, in which the diameter of the molten drop just breaking away from the electrode is 0.3 in. In normal arc melting practice, the arc gap is much smaller than in Fig. 2, and the length of time for a drop to fall through the arc gap is extremely short. Thus, there would be very little opportunity for any *thermal* purification in either the pool or the arc gap.

Vacuum factor

We may now examine the second mode of metal purification, namely *vacuum* purification. It can be said that the observed improvements in metal purity resulting from vacuum melting are due primarily to the attainment of a sufficiently high vacuum over the melt. The high degassing effectiveness of the electron beam melting process is now well established, and it is of interest that the principal developers of this process have recently stated[2] their conclusion that the metal purification attained in electron beam melting is due primarily to the unusually high vacuum achieved over the melt surface. Such a high vacuum is of course a prerequisite to the formation of the electron beam itself. This is not the case with arcs, which have always been more commonly operated in gas atmospheres than in vacuum. Existing vacuum arc furnace designs have been largely inherited from the inert-gas atmosphere furnaces of about ten years ago. These designs are still poorly advanced with respect to the important requirement of providing a really good vacuum over the melt. In fact, there appears to be no published evidence that a truly high vacuum over the melt in an arc melting furnace has yet been accomplished or even seriously attempted. On the other hand, electron beam melting furnaces have been designed intentionally to provide a high vacuum over the melt surface, and superior purification has been attained. An idea of the room for

improvement of arc melting in this respect is seen from Mr. Noesen's finding that the actual gas pressure over the melt in a vacuum arc melting experiment on molybdenum was 3000 μ, or 3 mm Hg.

We have been able in our laboratory to perform a considerable number of measurements of the actual gas pressure over the melt during the vacuum arc melting of iron and steel. Our results agree with Mr. Noesen's data on molybdenum in showing that the pressure over the melt is commonly several *millimeters* while the pressure near the vacuum pumps is only a few microns. In the vacuum arc remelting of a rotor forging steel we obtained pressures over the melt of 5 to 15 mm Hg. In the remelting of Armco iron we observed pressures of from 10 to 30 mm; so, it is obvious that there is still a long way to go in improving the vacuum arc melting process with respect to the important requirement of providing a satisfactory vacuum over the melt.

Improved performance

Mr. Noesen referred to two principal ways of improving the vacuum over the melt surface. One of these was to improve the crucible geometry so as to reduce the impedance to gas flow between the melt and the vacuum pumps. Our own experiments strongly confirm the effectiveness of this step. In the vacuum arc remelting of carbon-doped electrolytic iron, for example, we found that reducing the electrode diameter from 2 to 1.4 in. in a 3.5-in. diam crucible increased the degree of CO removal by factors of up to 2.6.

The second suggested way of improving vacuum purification was that of decreasing the electrode melting rate. Of the five ways Mr. Noesen listed, the method of increasing the electrode diameter is clearly in the wrong direction for improving purification. Varying the electrode density is feasible with powder-metallurgy electrodes but not with the cast or wrought electrodes commonly used in the arc remelting of steel. Adding certain gases may alter the electrode consumption rate, but the accompanying increase in the total gas pressure would probably have an adverse effect on the purification.

The effect of varying the crucible diameter is a very interesting phenomenon that Mr. Noesen is reporting here for the first time. However, it seems to apply only to tungsten and is certainly not a factor in the melting of iron and steel. Hence, the only one of the five ways listed for reducing the melting rate that would appear to have a chance of improving the purification of steel is the method of reducing the arc power. Mr. Noesen has provided some convincing data on this effect in the case of molybdenum. We have also tested this variable with iron, however, and were surprised to discover that it had no effect on the degassing at all. Carbon-doped electrolytic iron electrodes melted at a low arc power level were found to exhibit exactly the same degree of CO removal as those melted at a higher arc power level and at a much higher melting rate; so, there is a discrepancy in the experimental data on this point. In view of this, we must conclude that the only sure way of improving purification at the present time is by improving the geometry of the crucible-electrode configuration.

Arc cathode phenomena

For increased understanding and control of purification during vacuum arc melting, I suggest that we also examine some of the phenomena occurring at the arc cathode, or the *electrode*. One of these is metal spatter. Mr. Noesen suggested that the *skull* of impure metal surrounding the ingot is the product of successive evaporation and condensation of impurities from the pool. In the case of steel, however, we observe that this skull is formed principally from spattered molten metal emanating directly from the electrode. Molten metal drops forming at the electrode tip commonly become filled with gas (as bubbles) and burst, flinging molten drops against the crucible wall where they freeze to form the skull. The severity of this spatter increases with the amount of volatile impurities in the electrode. Relatively gas-free electrodes do not produce the spatter, and practically no skull forms. Mr. Noesen's observation that the skull has a high impurity content may be due to its being at an intermediate temperature and acting as a *getter* for volatile impurities evaporating from the melt.

Mr. Noesen mentioned that the principal arc cathode mechanism in the case of the highly refractory metals is thermionic emission, which usually requires a high surface temperature. There is reason to believe that the principal cathode mechanism of the steel-electrode arc is also thermionic emission. If so, a small cathode-spot area of the electrode would have to be highly superheated. This area should be found at the very bottom tip of the electrode, where the molten drops form and break away. Practically every bit of the material in the electrode must eventually pass through the stage of being a part of such a molten drop; therefore, the entire furnace charge would have the opportunity of being superheated, hence, thermally purified at the cathode.

We have observed that the melting of the electrode often takes place well up along its sides, as well as at the very bottom tip. The metal melting on the electrode sides forms a thin liquid film that flows continuously downward over the electrode surface to form drops that fall from the tip. This flowing molten film has an excellent opportunity to react chemically with its immediate environment, which is ideally a high vacuum. Thus, there is a good opportunity for both thermal and vacuum purification of the entire furnace charge at the cathode.

Conclusion

Vacuum arc melting of iron and steel is similar to that of refractory metals with respect to mechanisms of arc behavior and metal purification. Elimination of impurities due to strong superheating of the melt seems unlikely in the pool or the arc gap, but it may occur at the electrode tip. There is an opportunity for significantly improving purification by increasing the degree of vacuum over the melt and the electrode surface. This is best accomplished by reducing the gas-flow impedance of the crucible-electrode configuration.

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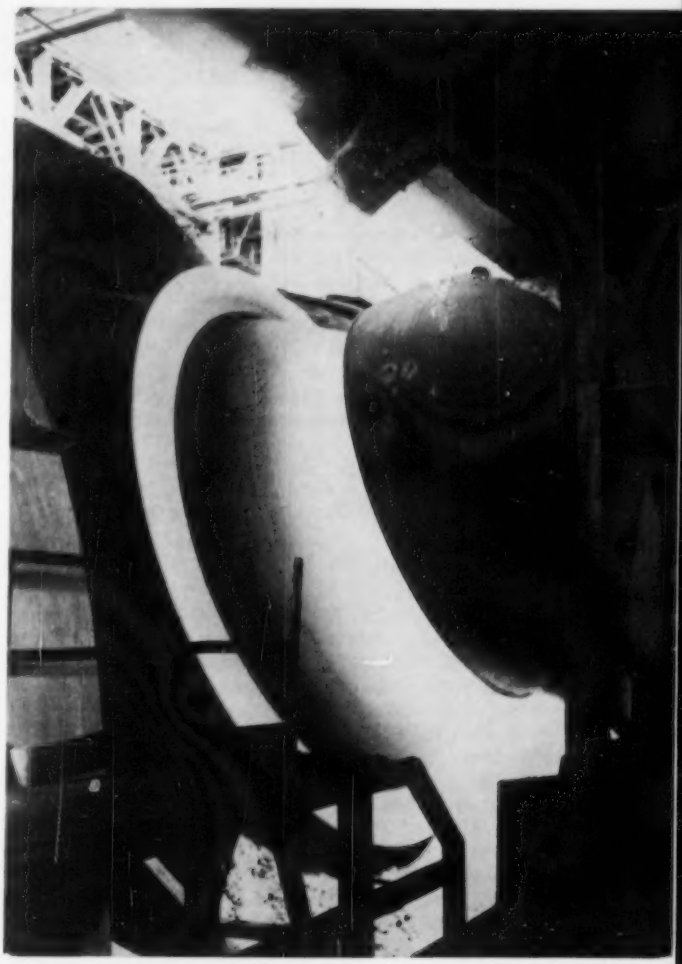
KALDO MOVES IN

A new 110-ton unit in France

Dramatic evidence that more than one oxygen steelmaking process is on the move is given by the successful completion of the first six months of Kaldo operations at the Sollac plant in France.

by F. Weston Starratt

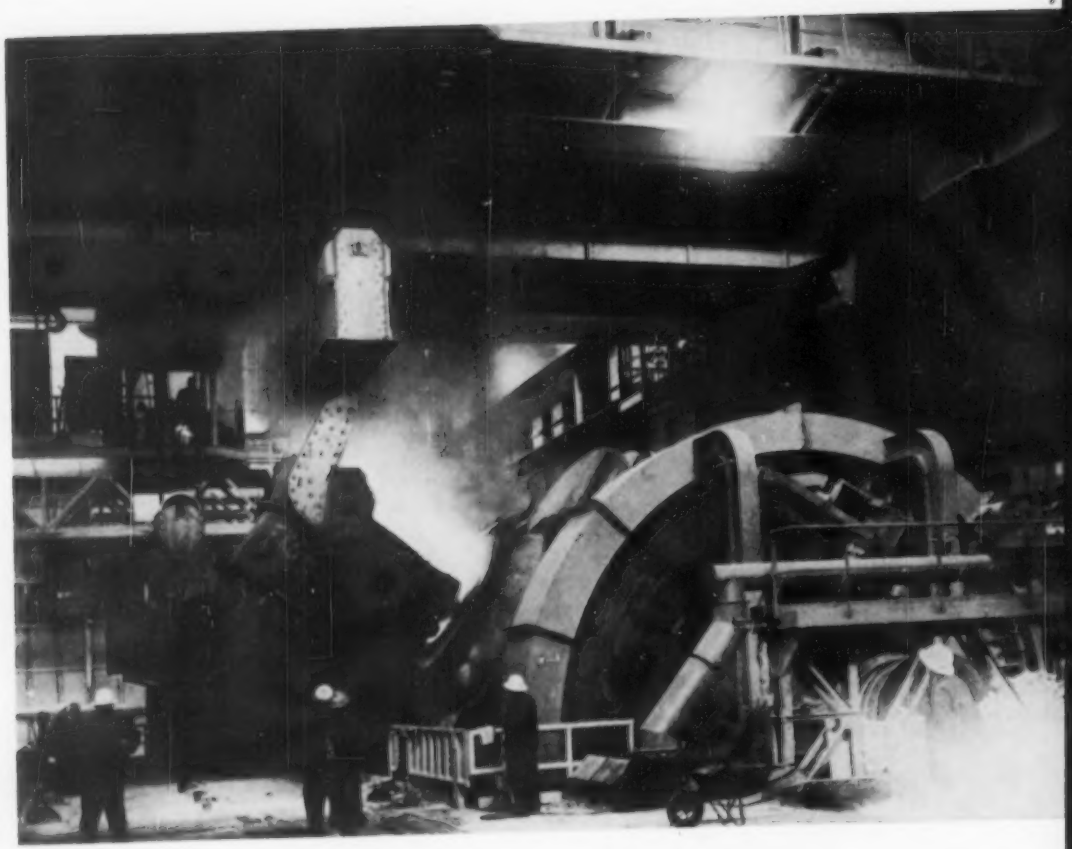


UNIQUE indeed—for there is probably no other enterprise like Sollac (Société Lorraine de Laminage Continu) among the world's steel plants, past or present. Sollac is a cooperative steel plant and rolling mill, lacking its own ironmaking facilities on the one end and its sales operations on the other. It is owned by surrounding ironmaking plants which supply it with hot metal brought in by torpedo ladle cars. These same firms take all of the output of Sollac and sell it through their own organizations.

Situated in heavily industrialized Lorraine in northeastern France, Sollac was organized more than 10 years ago to pool financial resources sufficient to construct—with Marshall Plan aid—the Continent's largest continuous wide-strip mill. To feed this 3 million ton a year mill, a steelmaking plant was built, and cold-rolling and coating facilities were installed to process the hot-rolled coils.

At the outset, Sollac's modern steelmaking facilities were unusual, for they combined four 60-ton basic bessemer converters—using an oxygen-enriched blow—with an open-hearth shop boasting two 155-ton and two 220-ton furnaces, designed to make use of scrap from the plant's rolling operations. Combined, these facilities were unable to furnish much more than 1.5 million tons of steel to the rolling facilities, and ingots and slabs had to be supplied by the participating firms. It was obvious that extension of the steelmaking facilities would be required.

Today an addition has been made—a 110-ton Kaldo furnace—thereby making this really a one-of-a-kind steelmaking unit, for where else in the world can one find these three steelmaking processes operating side by side under one roof? The Kaldo plant has been installed in an extension of



Charging hot metal.

the spacious open-hearth shop, with the basic bessemer plant in the opposite end of the building and the hot-metal mixers located between the basic-bessemer and the open-hearth shops.

There is a further unusual feature of this plant —this new addition is the first large scale industrial Kaldo furnace—a contrast to its 30-ton predecessor at Domnarvet—and parallel in size to the unit scheduled to go into operation at Oxelosund, Sweden, in 1961.

Why Kaldo?

While Sollac has increased its facilities for producing tinplate and electro-galvanized sheet, the greatest pressure on its production facilities has come in the area of cold-rolled sheet for the rapidly expanding markets for automobiles and consumer-durable goods. Therefore, expanded steelmaking facilities would have to be capable of producing the best quality deep-drawing steel. But for Sollac, the only economically available materials from which to produce this steel, are high-phosphorus hot metal obtained by smelting local Lorraine ores and scrap.

Sollac's basic-bessemer steel produced by blowing oxygen-enriched air, while adequate for many purposes, was not considered to be of sufficiently high quality for deep-drawing purposes; furthermore, supplies of hot metal are presently limited. This situation seemed to indicate an expansion of open-hearth facilities, but Sollac decided to study the possibilities of the available oxygen processes because of high raw-materials costs of the open-hearth steel—due to the utilization of hematite pig iron bought on the domestic market—and high operating costs—due to the cold charging practice required.

Thus, engineers of Sollac made a thorough investigation of all known processes for converting the Lorraine high-phosphorus hot metal into deep-drawing steel. Test runs were made at several steelmaking installations and pilot plants in various countries, and after considerable deliberation, Kaldo was selected as the steelmaking process most suitable to conditions at Sollac. Kaldo could produce the finest quality low-phosphorus steel from the high-phosphorus hot metal available at a lower cost than was possible in the other oxygen processes and, as mentioned before, in the cold-charged open-hearth furnaces. This was due to the fact that Kaldo can melt scrap—up to 40 pct of the weight of the steel produced—or smelt ore or combine both systems, thus showing a remarkable versatility, enabling it to follow the economic conditions. Furthermore, the process provided precise control over steel analysis, thereby almost eliminating off-grade heats.

Initial results

After six months of Kaldo operation, Sollac is more than satisfied with the results. Up to 40 pct scrap is being melted on a production basis, and the Kaldo steel is claimed to be the best deep-drawing steel produced by Sollac.

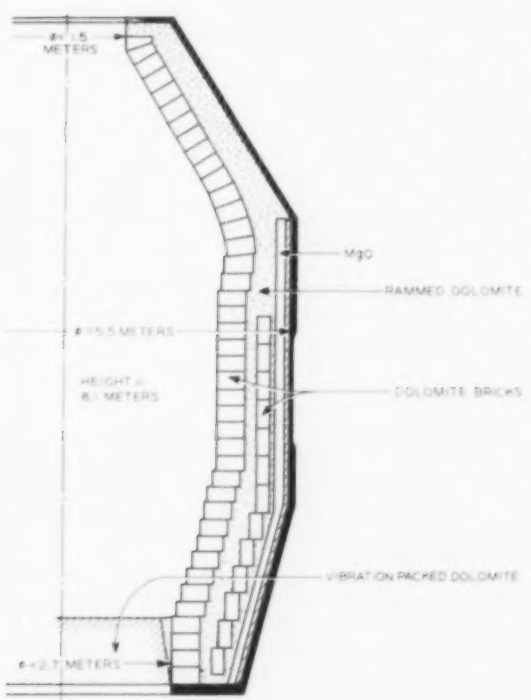
Both the open-hearth and the Kaldo furnaces are being used for the production of cold-rolled sheet. Steels containing less than 0.015 pct P can be produced by both processes, although the operation is longer and hence more costly in the open-hearth. Furthermore, it is considerably easier to produce steels of less than 0.015 pct S in the Kaldo than in the open-hearth, and open-hearth steels at Sollac average 0.004 pct N, while Kaldo steels analyzing 0.002 pct N are being produced.

Sollac's Kaldo steel is being produced from hot metal of the following analysis: 3.5 pct C, 0.35 pct Mn, 1.75 pct P, 0.040 pct S, and 0.5 pct Si. Thus far, Kaldo steel has been produced according to the following average analysis: 0.05 to 0.07 pct C, 0.014 with a 0.017 max pct P, 0.250 to 0.400 pct Mn, and 0.012 with a 0.018 max pct S. Killed, capped, and rimming grades have been produced. Ingots, which range from 10 to 13 metric tons, are top poured.

The installation

The Sollac installation consists of two interchangeable 110-ton Kaldo furnaces and one blowing stand designed and built by the Demag firm of Germany and Compagnie des Ateliers et Forges de la Loire of France. The total weight of the furnace shell with running rings is 196 tons, to which should be added about 300 tons for the lining. Maximum rotation speed of the furnace is 30 rpm, and the inclination of the vessel during the blow is 17°. The furnace can be removed from the rotating stand by a transfer car provided with a hydraulic lift.

Kaldo furnace lining.



Scrap is charged with tilting boxes which are maneuvered from the control room on the tapping pulpit. The hydraulic system for tipping the boxes is built into the platform on the tapping side. There are two scrap cars, each with two boxes which can hold some 10 tons of billet scrap, but only 3 tons of scrap bundles.

Lime and fine ore are transported to bunkers above the blowing side of the furnace by an overhead trolley which serves the whole steel plant. There are four lime bunkers and two ore bunkers, as well as weighing bunkers. There are two additional bins: one holding lime for the tapping dam and the other coke for burning in.

Oxygen of 99.5 pct purity is supplied at a pressure of 43 psi from a plant capable of producing 260 tpd, a quantity sufficient for enriching the blast of the basic-bessemer converters and a limited Kaldo production but not for basic-bessemer and Kaldo operations on a three shift basis. Additional oxygen supplies are forthcoming from a recently-constructed regional oxygen plant.

The water-cooled oxygen lance consists of three concentric copper pipes, the inside pipe of which is narrowed at the mouth to 110 mm with an expanding nozzle. The lance can be moved axially and is held in place on a sloping track with an electric hoist. The slope of the track can be changed with a set screw and also with an eccenter. The latter is motor-driven in order to give the oxygen lance a sweeping motion up to a maximum of 10°.

For dust removal, a Pease-Anthony venturi washer of design similar to that at the Bochum, Germany, LD steel plant has been installed (see JOURNAL OF METALS, July 1960, pp. 554-557). Some 10 lb of dust per ton of steel is removed from the fumes, leaving the escaping gas with less than 100 mg of dust per cu m.

Furnace lining

During these early months of operation, Sollac has been blowing its Kaldo furnace on a one-shift-per-day basis, with the furnace being allowed to cool during the remainder of the day. This is only a temporary state, and a burner for keeping the furnace warm during this period has not been put in service. The result is that for the first blow of the operating shift, hot metal is poured into a cold furnace. This is obviously not good for refractory life, and gross refractory consumption now is 44 lb per ton, of which 8 lb is recycled, thus making 36 lb net.

The lining was found to be able to withstand from 70 to 100 heats, but up to this time each campaign has been stopped after about 60 heats. It has not yet been possible to attain the optimum relining schedule; so a furnace may be relined and ready for service before lining wear would necessitate replacing the operating furnace. Furthermore, the mechanism designed to provide the oxygen lance with a sweeping motion has not yet been installed: so, excessive lining wear may now be experienced in one particular ring area. The result of these factors is that both the figures for refractory consumption and lining life must be considered as only an indication of better results to be achieved in the near future.

Relining now requires a period of six days, followed by a baking period of up to 12 hr, after which the furnace must be placed in service as soon as possible. An accompanying cross-sectional diagram shows the details of the masonry work on the Kaldo furnace.

Tap-to-tap

In discussing the present operating cycle for the Kaldo furnace at Sollac it should be emphasized that this is a new plant and that many features of the operation have not yet been fully coordinated and put into service. Thus far, the furnace has been operating on the basis of a 2 hr tap-to-tap schedule with a slightly longer schedule for the first blow of the shift in a cold furnace. But ancilliary installations still to be installed and changes in operating practice should bring this time down to at least 1 hr 45 min and possibly 1 hr 30 min under optimum conditions.

Heavy plant scrap has, thus far, not been available in sufficient quantity to permit charging with only this type of scrap, and a large portion of the charge has been made up of lighter scrap bundles. By charging only heavy scrap a saving of 15 min could easily be made in the present cycle. It should be noted that by charging ore instead of scrap this tap-to-tap time could be even further reduced.

A second important factor which should permit more rapid operation is the change-over in method being used for charging lime and cooling ore. Installation will soon be made of ore and flux chutes which will permit continuous feeding during the blow rather than the present system of stopping the blow, raising the furnace, and charging these materials from overhead chutes.

On the tapping end, improvement will be made when an apparatus is installed to make lime additions to solidify the slag and create a slag dam. Presently, this is a hand operation. Improvement is also anticipated in the actual blowing time. It now averages 70 min, but 60 min blows have been achieved, and this may become the norm in several months.

The cycle

The following is the operating cycle developed during these first few months of producing low-carbon deep-drawing steel in the Kaldo furnace:

1) With about 3 tons of partially-solidified slag from the previous heat, some 80 tons of hot metal at 1200° to 1250°C is charged into the furnace. Average analysis of the hot metal for the first four months of operation is: 3.5 pct C, 0.35 pct Mn, 1.75 pct P, 0.040 pct S, and 0.5 pct Si.

2) One half—25 tons—of the required scrap is charged, and the furnace is turned up for the addition of about 7 tons of lime.

3) The furnace is then blown with about 35,000 to 53,000 cu ft of oxygen to melt the scrap.

4) The second 25 tons of scrap is charged.

5) The furnace is again turned to the blowing position and the operation proceeds as careful watch is kept on temperature measurements in the exhaust stack. The temperature begins to drop as dephosphorization starts, and then another 35,000 cu ft of oxygen is blown before deslagging, making a total of 195,000 to 205,000 cu ft of oxygen blown from the beginning. At this point the metal has attained the following composition: 0.350 pct C, 0.200 pct Mn, 0.035 pct P, and 0.020 pct S.

6) The furnace is turned down for removing a slag containing 18 to 20 pct P_2O_5 and 6 to 7 pct Fe. A metal sample is taken for analysis, and the bath temperature—which is generally about 1580°C—is measured by an emersion thermocouple.

7) The final phase of the blow starts with the addition of about 2 tons of lime and sufficient quantity of fine ore to cool the bath to the desired temperature. When it is felt that the desired metal analysis has been attained, the furnace is turned down, a metal sample is taken and analyzed, and—if correct—sufficient lime is added to form a slag dam, and the metal is poured. The average metal analysis obtained after addition of deoxidizing agents in the ladle is the following: 0.05 to 0.07 pct C, 0.280 to 0.400 pct Mn, less than 0.017 pct P, less than 0.018 pct S, and about 0.002 pct N.

Oxygen is now being blown at the rate of 3900 cu ft per min, although at a later point when the lance has been provided with equipment to give it a sweeping motion, blowing may be stepped up to 5000 cu ft per min, at least on a trial basis. About 260,000 cu ft of oxygen are blown per heat or 2100 cu ft per ton of steel.

The results of the first four months of operation show an average of 125 tons of steel produced per heat, some 15 pct above the rated capacity. The charge has been made up of an average of 61 pct hot metal and 39 pct scrap. Some 200 lb of lime have been used per ton of steel. Ingot yield (weight of ingots divided by weight of hot metal, scrap, alloys, and Fe in ore) now averages between 90 and 91 pct.

Laboratory control

Rapid and accurate analysis during the melting cycle is essential for the Kaldo process. It is for this reason that installation of a Quantovac vacuum spectrograph is being made at a location adjacent to the melt shop. This equipment and its location should permit a metal analysis to be made within 2 min and thus promote some acceleration of the present steelmaking cycle.

In order to ensure a homogeneous sample for spectrographic analysis, metal samples are taken by plunging into the metal bath a closed steel mold inclosing an aluminum wire and capped with a small piece of wood. Steel within this mold is killed with the aluminum wire and removed after solidification.

A carbometer is located in the furnace control room to permit rapid checking of the final carbon content before tapping.

Conclusions

The first six months of operation of the 110-ton Kaldo furnace at the Sollac plant in northeastern France have shown that it is capable of producing higher quality low-carbon deep-drawing steel at a lower cost than is possible with the plant's open-hearth furnaces.

Three-shift-per-day operation is being planned in the near future, and this factor, together with the installation of certain ancilliary equipment and increased operating experience is expected to reduce tap-to-tap time, increase refractory life, and result in an even more efficient operation.

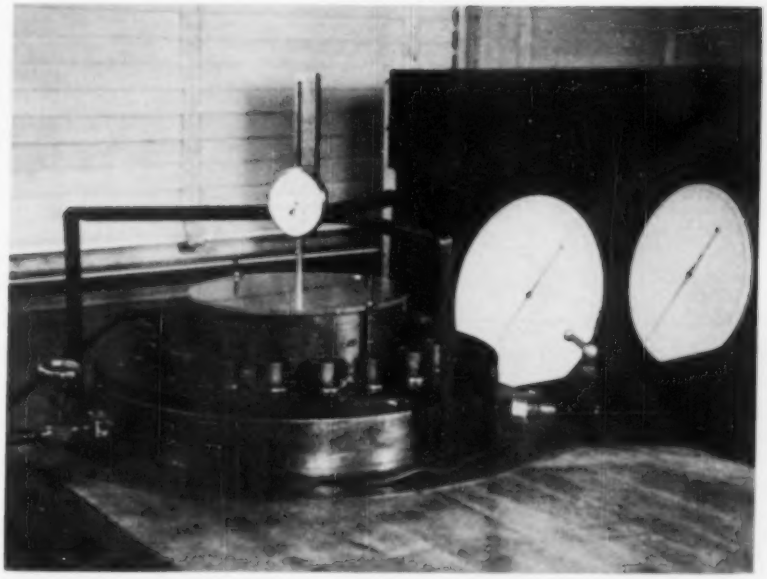


Fig. 1

HYDRAULIC BULGE TESTER

DETERMINES SHEET DRAWING QUALITY

To date there has been no completely satisfactory method for determining the drawability of a sheet. Employing a hydraulic bulge tester, it is possible to utilize the instability point of a metal under biaxial tension as a sensitive criterion of drawability.

by

W. N. Lambert,

E. S. Madrzyk,

and F. E. Gibson

There is no completely satisfactory method of determining the drawability of a sheet; therefore, much effort has been expended to find a method that would adequately measure this property. Many tests have been proposed and devised to obtain an indication of the probable behavior of a sheet under a press; these include simple bend tests, slotted-strip tests, tear-length tests, mechanical and hydraulic cupping tests, and tensile tests.[1] None of these tests, however, seem to provide an accurate index of drawability when compared to actual press performances.

W. N. LAMBERT, E. S. MADRZYK, and F. E. GIBSON are with the Research and Development Dept., Inland Steel Co., East Chicago, Ind. This paper was presented at the 1959 Fall Meeting of The Metallurgical Society.

Table I. Tabulation of Various Index Values for Nonferrous Metals and Intermediate Temper Steel

Sample	Grade	Gauge	h Value	Corrected h Value	n[a]	Olsen	Rn
Brass	65-35	0.0314	3.83	3.92	0.540	0.452	22.8
½ Hard Brass	65-35	0.0180	3.48	3.66	—	0.319	62.7
Copper	Commercial	0.0325	3.28	3.38	0.340	0.385	53.3
Aluminum	52 S	0.0307	3.19	3.30	0.280	0.308	57.5[b]
Aluminum	2 S	0.0323	3.14	3.24	0.250	0.334	35.3[b]
Steel[c]	Killed	0.0359	2.96	3.04	—	0.325	72.0

[a] Values taken from reference No. 4.
[b] Rockwell 15 T scale reading.
[c] Intermediate temper.

The Olsen cupping test has evolved as a standard qualitative test of drawing quality in mills, because of its simplicity and rapidity of operation. Mechanical cupping tests would undoubtedly provide a truer index of drawability, if it were not for several inherent faults, two of which are 1) the friction between the punch and the test piece and 2) the smallness of the area tested.

About 1930, Jovignot[2] proposed the fluid cupping test, in which a liquid is substituted for the metal punch in an effort to eliminate friction between the punch and test piece. He suggested that the height of the bulge and the pressure at failure be measured and that a *cupping coefficient* $= \frac{(\text{height of bulge})^2}{(\text{radius})^2}$ be used as a criterion of formability. Succeeding workers proved that this coefficient could not discriminate between good and poor drawing quality.

The tensile test has been used as one of the laboratory methods of determining the drawing properties of a sheet. Several investigators have shown that true-stress true-strain for most metals closely obey the equation, true-stress $= K(\text{true-strain})^n$. The coefficients K and n are referred to as the strength coefficient and the strain hardening exponent, respectively, and vary with the material. It has been shown mathematically[3] that the slope of the true-stress true-strain curve, the n value, is numerically equal to the strain at which necking begins. This value can be considered the instability point, i.e., the point at which flow proceeds at constant load, ($dL = 0$), for the material under uniaxial tension. Arbel[4] showed that the n value can distinguish between materials of very different drawing quality. Using hard brass, aluminum, copper, soft brass, and stainless steel, he obtained a range of n values from 0.07 to 0.54 for these metals in the order listed. These correspond very well to the drawing value of each material as obtained from the *Tinius Olsen* cupping tests*. Lankford, Snyder, and Bauscher[5] suggested using an R value combined with the n value as a criterion of drawing quality. The R value is defined as the ratio of the width strain to thickness strain and is a measure of the anisotropy of the sheet. Using 46 lots of steel, they show fair correlation between the n and R values and actual press performances.

Many investigators[2,6,7,8,9,11,12] have mathematically analyzed the stress-strain relationships involved in a hydraulic bulge test and have derived expressions that conform very closely to observed data. Several

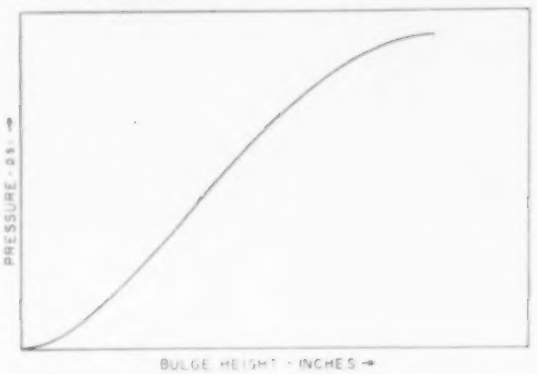


Fig. 2—Typical bulge curve obtained from all metals.

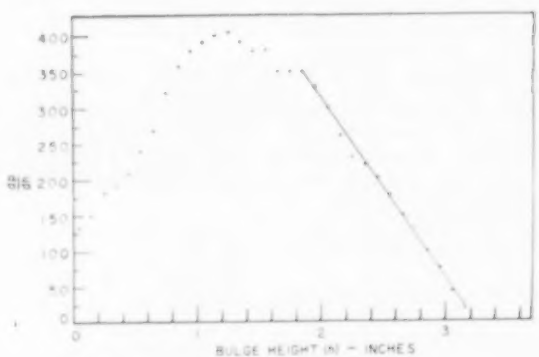


Fig. 3—Parabolic nature of upper portion of bulge curve.

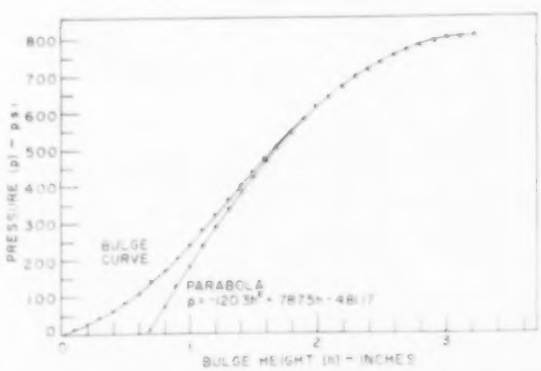


Fig. 4—Agreement between upper portion of bulge curve and corresponding parabola.

* This test comprises the successive drawing of specimens of increasing diameter through a fixed diameter die and calculating the maximum diameter of the specimen which can be drawn. The ratio $\left(\frac{\text{maximum blank diameter}}{\text{punch diameter}}\right)$ is used as an index of drawability.

workers[5,6,9,11] have shown that it is possible to obtain true-stress true-strain curves from bulge data that correspond very closely to those obtained from tensile data through the use of octahedral or effective shear stress and shear strain. Lankford[5] measured increments of strain at the pole of the bulge. Epstein and Frame[11] were able to distinguish between a poor and a good lot of drawing quality steel, having similar physical properties, by measuring the uniform elongation in a 6-in. circle at the top of the bulge.

A bulge tester, similar to that used by Lankford and others[5,6,8,9,10,11,12], was designed and built with a 10-in. diam bulge opening. Fig. 1 shows the equipment in operation. Isenberg[12] derived theoretical equations relating to bulge and tensile tests, incorporating only the simple measurements of height of bulge and pressure. During the progress of the test, these two parameters were measured simultaneously.

The machine was tested for reproducibility. Duplicate tests were run on various gauges and grades of sheet steel, and it was found that the bulge results were reproducible within the limit of error of the measuring instruments. Thielsch[13] has shown that, in comparison with tensile tests, bulge testing gives results of far greater reproducibility.

Correlation with Drawability

The first approach was to correlate the bulge data with the n, R, and K values of the tensile tests. Isenberg[12] derived theoretical equations to obtain n, R, and K values directly from the bulge data. He suggested obtaining a family of curves to do this. It was found that an infinite number of tests would be required; therefore this approach was rejected. Isenberg then suggested that the log-log plot of the lower portion of the bulge curve would give the n value directly. This was checked with actual data but was not borne out. During the compilation of press performance and tensile data, it became apparent that little, if any, correlation existed between the two. In view of this lack of correlation, any further attempts to obtain tensile values from the bulge test did not seem warranted.

Efforts were then directed to finding a criterion of drawability from the bulge data per se. Numerous attempts were made to obtain a general equation for the bulge data when pressure was plotted against the height of bulge as shown in Fig. 2. To determine the type of curve represented by the bulge data, various plots were made, which included semi-log, log-log, and probability plots. Attempts were also made to draw an analogy with the tensile method of calculation using increments of increase in strain versus load. The conclusion was that the entire bulge curve could not be represented by a simple function.

It had been suggested that an instability point be used as a criterion of drawability. Lankford[5] pointed out that this is the point where $dp = 0$ with increase in strain, where p stands for pressure. This is the value for biaxial tension analogous to the n value ($dL = 0$ with increase in strain) for uniaxial tension. Since drawing operations are biaxial, the instability point in the bulge test would be an accurate criterion of drawability. Thielsch[13] showed that the circular bulge test is considerably more sensitive than the conventional tensile test to variations in sheet specimens. The instability point on the pressure-height curve of each steel was determined

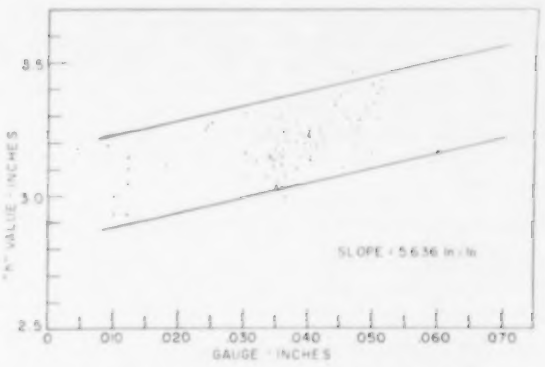


Fig. 5—Gauge correction plotted from steels bulged.

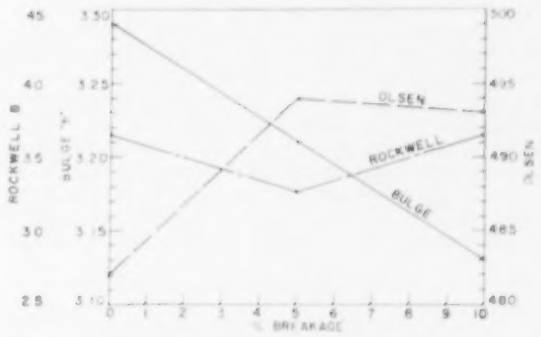


Fig. 6—Trailer bathtub physical properties.

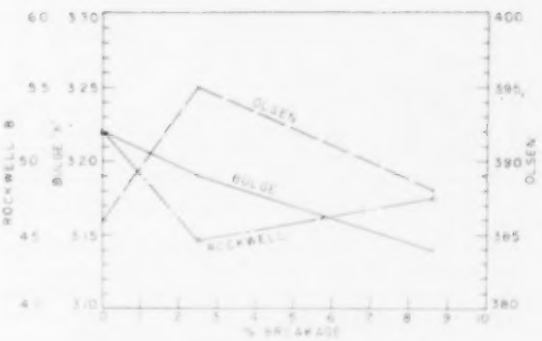


Fig. 7—Rear quarter outer physical properties.

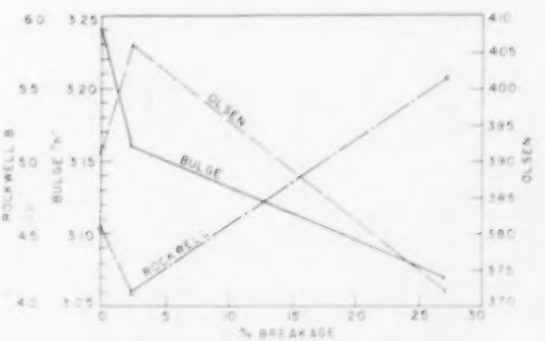


Fig. 8—Door outer physical properties.

roughly by a graphical method. The instability point so determined was termed the *h* value of a particular steel. The inaccuracy of this method precluded the precise location of the point. However, the spread in these points was sufficient to indicate that a discriminatory index of drawability could be obtained, if a more precise positioning of the point were possible.

It was noted that, if the first derivative $\frac{dp}{dh}$ could be obtained for the bulge curve, the point at which this slope became zero would be the h value. Since the equation of the curve was not determined, mechanical differentiation was used. A plot of $\frac{dp}{dh}$ versus height of bulge revealed a reasonable straight line over the portion of the curve that represented the upper portion of the bulge curve as shown in Fig. 3. This clearly indicated that this portion of the curve was a parabola. The method of differences was employed to obtain the general equation of the parabola which turned out to be of the form $p = C_1 h^2 + C_2 h + C_3$. As a final check, an experimental bulge curve and the calculated corresponding parabola were plotted on the same graph. The agreement was excellent, as shown in Fig. 4.

The h value was determined by differentiating the equation $p = C_1 h^2 + C_2 h + C_3$, setting the first derivative equal to zero and solving for the maximum on the h axis. This gives the equation $h = \frac{-C_2}{2C_1}$. In order to evaluate the constants, three standard height of bulge points were chosen in the following manner: first, the highest possible 0.1-in. bulge height reading from the data was chosen; then, a second point was selected to be 0.4 in. less than the first point; and finally, the third was chosen to be 0.4 in. less than the second point. In all instances this seemed to keep the chosen points within the parabolic range.

The corresponding pressure values were read from the actual bulge data, and these three sets of points were then substituted into three parabolic equations having three unknown constants. The h value was then determined by the use of one determinant.

Fig. 5, where h values are plotted against the corresponding gauge, shows that, in order to compare the drawing quality of steels of different gauges, a correction factor of 5.636 in. per in. of gauge must be employed. Subsequently all h values have been corrected to a standard gauge of 0.050 in.

Table II. Tabulation of Index Values and Press Performance for Various Parts

Coil	Gauge, In.	Pct Breakage	h Value	R Value	n Value	Olsen	Rn
Teletype Cabinet Top							
5637	0.0525	1.20	3.43	1.89	0.253	0.421	36.5
5638	0.0505	1.31	3.41	1.82	0.254	0.423	37.5
5641	0.0500	0	3.35	1.81	0.258	0.421	36.0
5640	0.0500	37.7	3.28	1.78	0.257	0.423	37.5
Oil Pan							
5945	0.0500	0.10	3.43	1.80	0.256	0.402	37.5
5948	0.0515	0.16	3.39	1.78	0.268	0.414	40.0
5947	0.0505	0.21	3.32	1.77	0.257	0.401	38.5
5946	0.0510	0.78	3.15	1.48	0.231	0.399	39.0
Upper Shroud Panel							
33	0.0387	0	3.40	1.86	0.269	0.443	39.5
35	0.0359	0	3.37	1.81	0.258	0.435	36.0
36	0.0368	0	3.35	1.84	0.269	0.431	36.5
29	0.0356	0	3.32	1.83	0.248	0.441	38.5
30	0.0353	0	3.27	1.87	0.270	0.436	37.0

Validity of Index

To test the validity of the drawability index over a wide range of drawabilities, samples of various nonferrous metals and intermediate tempered steel were tested. The results, shown in Table I, indicate that correct rank order of drawability is easily established by the bulge test.

To test the discriminatory power of the bulge index, where small variations in drawing quality are encountered, actual press performance data was collected. These data, along with corresponding R and n, Olsen and hardness values, are compared in Table II. This table shows that the bulge index is sensitive to small variations in drawing quality. In all but one instance, the h values are in the same rank order as the press performance. The n and Olsen values have no discriminating power upon this narrow range of drawing qualities.

These data substantiate previous findings that the bulge index has fine discriminatory powers. To further illustrate this data, Figs. 6, 7, and 8 show the tests results plotted against performance for three parts where there were three levels of performance. It is evident that the difference in performance cannot always be determined by Olsen and Rockwell tests.

Conclusions

1) The upper portion of the bulge curve can be represented by the general parabola equation, $p = C_1 h^2 + C_2 h + C_3$.

2) A method has been developed to determine accurately the instability point for biaxial tension.

3) An index of drawability with fine discriminatory powers and reliability has been developed.

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HIGH-PURITY ELECTROLYTIC CHROMIUM

A study of massive chromium electrodeposited from aqueous chromic-acid electrolytes at elevated temperatures—the relative effect of the catalytic action of sulfate, fluoride, and silicofluoride ions on mechanical properties was investigated with a subsequent determination of optimum compositions.

by M. J. Ferrante, P. C. Good, F. E. Block, and D. H. Yee

The principal object of this investigation was to prepare kilogram quantities of high-purity chromium by the electrolysis of chromic acid solutions. The impetus for this investigation arose from the recent attention that has been focused on the outstanding properties of chromium. Its high resistance to corrosion, oxidation, and abrasion at elevated temperatures is of great interest to fields such as aviation and ballistics. Since no single investigation could study the many aspects of preparation, properties, and applications, this work represents only a part of a larger study on the physical and alloy characteristics of chromium. Details of these studies have been reported separately[1-4].

Of the various methods for preparing metallic chromium, electrolysis of chromic acid solution was selected because of the relative ease with which metallic impurities could be eliminated. High temperature baths are used because it has been demonstrated[5, 6] that electrolysis at elevated temperatures produces chromium of lower oxygen content.

For practical reasons, an effort was made to obtain the chromium metal at higher current efficiencies than the 8 to 10 pct values generally reported for the electrolysis of chromic acid-sulfate solutions. Some investigators have suggested that any large increase in current efficiency was unlikely[7] because of hydrogen evolution during the electrolytic reduction of hexavalent chromium[8]. Nevertheless, it was hoped that a significant improvement could be made by determining the optimum conditions for those variables that affect current efficiency. It was later found that much higher current efficiencies could be obtained by using the fluoride ion as a catalyst in the place of the sulfate ion. Substitution of the silico-fluoride ion for the fluoride ion resulted in an additional slight increase in current efficiency.

For the sake of brevity, the expressions *chromic acid-sulfate electrolyte* is replaced by the term *sulfate electrolyte*. Similarly, the term *fluoride electrolyte* and *silicofluoride electrolyte* refer to chromic acid baths to which these ions have been added.

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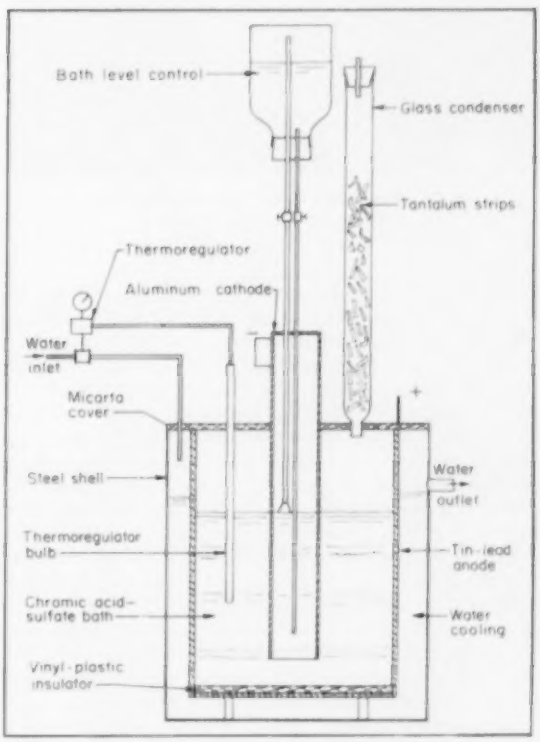

Fig. 1—Electrolytic cell for sulfate baths.

Sulfate Electrolytes

Equipment and Procedure

In earlier work, the electrolytic cell was a circular steel tank 18 in. in diameter and 16 in. in height. The tank was lined with unplasticized polyvinyl chloride which successfully withstood the operating temperature of 90°C. The anode consisted of a 16-in. diam coil of ¾-in. lead tubing which served also as a cooling coil. In later work, the cell (Figure 1) comprised a tin-lead alloy vessel 16 in. in diam and 15 in. in height set in a larger steel tank. The cell

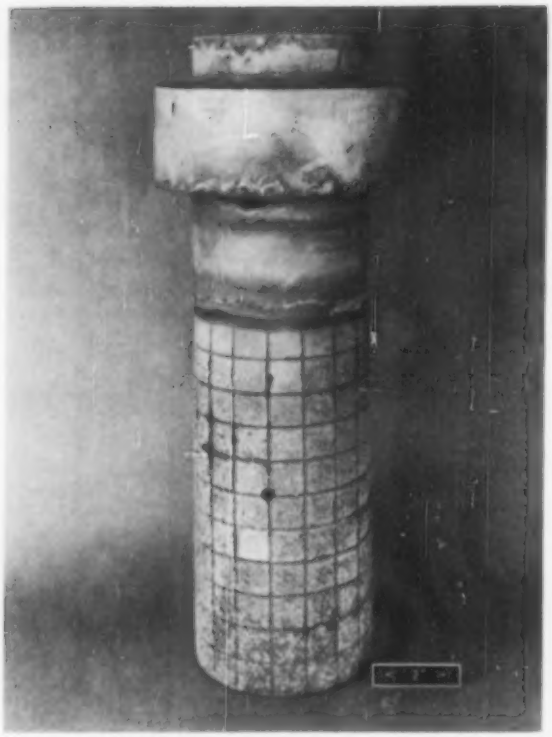

Fig. 2—Aluminum cathode used in sulfate baths.

capacity was 30 liters, with the cell wall serving as the anode. Circulating water in the annular space between the cell and the steel container controlled the electrolyte temperature. The original tubular lead anode was more effective in controlling the bath temperature, but, on the other hand, the pot-type anode suffered less corrosion. Ten percent tin-lead alloy was preferable to pure lead as an anode material because it withstood corrosion better. In either type of cell the flow of water was regulated with a Powers regulator activated by a temperature-sensitive, lead-sheathed bulb in the electrolyte.

A Micarta cover was fitted over the cell to direct escaping vapors to a glass reflux condenser loosely packed with pieces of tantalum sheet. Liquid losses were replenished by a reservoir placed above the cell and connected to a constant-level device to maintain a uniform electrolyte in the cell.

An aluminum pipe 6 in. in diam and 20 in. in length in the center of the cell served as the cathode. Fig. 2 shows that scoring the surface of the cathode into 1-in. squares caused a thinner deposit to be formed at the score marks and facilitated removal of the deposit. The deposit was removed by striking the cathode a sharp blow with a hammer. Direct current was supplied to the electrodes from a 4000-amp capacity selenium rectifier.

The electrolyte was prepared by dissolving technical-grade chromium trioxide in distilled water and subsequently adding sulfate ion in the form of sulfuric acid. During electrolysis the chromium concentration was determined twice daily by specific gravity measurements and was adjusted by additions of chromium trioxide either to the feed reservoir or directly to the cell. The sulfate ion concentration was adjusted at the beginning of each run on the basis of analysis. Prior to each operation, hot water was circulated around the cell to bring the electrolyte temperature near the boiling point.

The accumulation of ion impurities in the electrolyte in successive runs was prevented by passing the solution through a bed of cation exchange resin. Of several resins tested among the sulfonated polystyrene type, (which are recommended[10-12] for treating strongly oxidizing solutions), Amberlite IR-120 was the most resistant to oxidation in solutions containing up to 250 g per liter of chromic acid at room temperature. A resin bed 4 in. in diam and 30 in. in depth was employed. Analysis of the 10 pct sulfuric acid solution, used to regenerate the resin, showed that Al, Cu, Fe, Ni, Si, and Sn were being removed from the electrolyte.

Results and Discussion

It was found that a deposition interval of 72 hr produced an adherent deposit between 1/16 and 3/32 in. thick. In addition, a large portion of the deposit was in the form of tree-like growths or dendrites, which broke from the cathode during electrolysis and dropped to the bottom of the cell. Although the dendrites could be recovered as metal, they contained consistently more oxygen than the deposit that adhered to the cathode. It was desirable, therefore, to establish operating conditions to suppress dendrite growth. The changing of cathodes at shorter deposition intervals was successful to some degree in minimizing dendrite formation, but the resulting thinner deposits were extremely difficult to remove from the cathode.

Table I shows the results of representative deposition tests conducted for 72- and 96-hr periods at cathode current densities ranging from 250 to 1100 amps per sq ft and at various bath compositions. Because the results in this phase of the investigation have been reported in detail[14, 15] only a brief discussion of the conclusions is given below.

The data obtained for the sulfate electrolyte established the following set of operating conditions which yielded maximum cathode current efficiencies while minimizing dendrite formation:

Current density	below 500 amp per sq ft
Chromic acid concentrations	125 g/l
Ratio: CrO_3/SO_4	50
Operating Temp.	85-90°C

Maximum current efficiencies of 12 to 13 pct were obtained when electrolytes were used having CrO_3 to SO_4 ratios of approx 50; somewhat lower efficiencies resulted when lower or higher ratios were employed.

The solid deposit removed from the cathode usually contained between 50 and 200 ppm O_2, less than 20 ppm N_2, and trace amounts of Al, Fe, and Si. Arc-melting reduced the hydrogen content of as-deposited metal from as high as 900 ppm to between 1 and 10 ppm. In all instances the dendritic deposits contained a significantly higher amount of oxygen than the corresponding adherent deposit. There was no detectable difference between dendritic or adherent deposits with respect to the amounts of metallic impurities present. Fig. 3 shows photomicrographs of arc-melted solid deposit and its corresponding dendrites electrodeposited from the sulfate electrolyte. The increased amount of sec-

ond-phase material in the dendrite deposit illustrates the higher oxygen level in this material.

Fluoride and Silicofluoride Electrodes

Although some improvement in current efficiency was noted by varying the plating conditions in the conventional sulfate electrolyte, a review of the literature disclosed that much higher efficiencies could be obtained with fluoride or silicofluoride baths[16]. Ryan[17] has reported on an extensive investigation of chromic-acid electrolytes containing fluoride ion operated at the boiling point. Because of the high current efficiencies of 40 to 45 pct obtained by Ryan, the preparation of chromium from fluoride electrolytes was investigated in this laboratory. This work produced somewhat lower current efficiencies than experiments conducted by Ryan. Noting that Ryan used glass equipment, whereas the authors employed tin-lead apparatus, tests were made to determine whether silica pickup from glass might have an effect on current efficiency. This problem was investigated by adding silica to the electrolyte as the silicofluoride ion.

Equipment and Procedure

Although the apparatus employed for the fluoride and silicofluoride baths was similar in general to that described for the sulfate electrolyte, certain differences in construction were necessary.

Fig. 4 shows the cell used; it had a capacity of 11 liters, a diam of 10½ in., and a height of 11 in. The upper edge was provided with a flange which was sealed tightly to a rigid, unplasticized polyvinyl chloride cover that directed escaping vapors to glass reflux condensers.

The cathode was a thin-walled copper tube 2½ in. in diam which had the lower end sealed off with a Teflon plug. The upper part, immediately above the electrolyte level, was protected from corrosion by polyethylene or Tygon tubing. The deposited chromium was separated from the copper cathode by dissolving the latter in dilute nitric acid. Aluminum cathodes corroded and yielded very poor results.

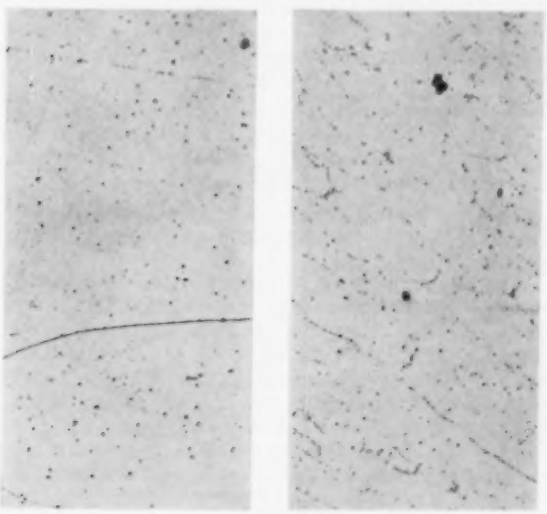

Fig. 3—Photomicrographs of arc-melted, as-cast chromium electrodeposits, prepared by chromic acid-electrolytic etch, 100X. Left, solid deposit, 40 ppm oxygen. Right, dendritic deposits, 600 ppm oxygen.

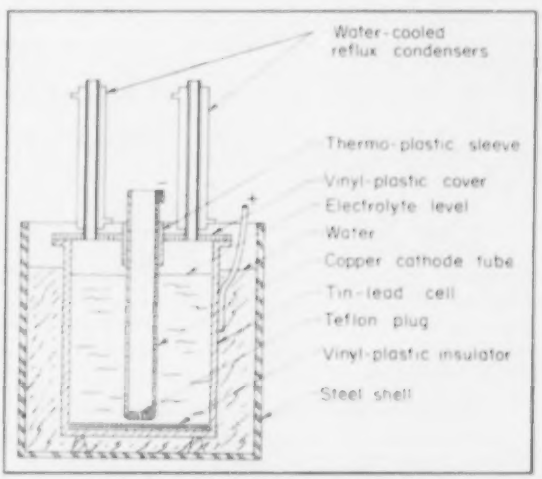

Fig. 4—Electrolytic cell for fluoride-type baths.

Table I. Results of Sulfate Electrolytic Tests

Run No.	Hrs.	Bath composition SO_4, g/l	Bath composition CrO_3, g/l	Bath composition Ratio CrO_3/SO_4	Cathode current density amp/sq. ft.	Cathode current efficiency, pct	Dendrite deposit, pct	Deposit type[a]	Analysis, ppm[b] O_2	H_2	N_2	C
1	72	1.8	115	64	250	12.8	1	P	30	1	10	50
3	96	1.0	125	125	350	7.7	0	P	50	1	20	80
5	96	2.1	125	60	365	11.6	6	P	70	2	10	50
7	72	2.5	125	50	386	13.6	3	P	200	2	10	50
9	72	2	125	63	515	12.7	28	P			30	
11	72	5.2	250	48	580	8.8	16	P	140	2	<30	
13	72	7.8	250	32	610	8.4	18	P	50	<1	<30	30
15	72	2.5	250	100	750	8.6	36	P	120	5	<20	
								D	590	5	<20	
17	72	2.5	250	100	750	11.2	33	P	250	4	<20	
								D	1280	8	30	
19	76	2.5	250	100	800	8.7	53	P	150	934	<20	740
								D	970	<20	<20	160
21	72	2.5	250	100	800	8.5	51	P	120	<20	<20	<50
								D	280	<20	<20	<50
23	72	2.5	250	100	800	7.3	54	P	200		<20	<50
								D	920		<20	<50
25	72	2.5	250	100	900	12.8	64					
27	72	2.5	250	100	1100	8.8	65					
29	72	2.5	250	100	1100	8.5	69	P	230	5	<20	
								D	1140	10	<20	

[a] P = deposit adhering to cathode; D = dendrite deposit fallen from cathode.
[b] Oxygen and hydrogen determined by vacuum fusion; nitrogen by vacuum fusion or Kjeldahl analysis.

Table II. Results of Fluoride and Silicofluoride Electrolytic Tests

Run No.	Hr	Bath composition: HF, F g/l	Bath composition: H_2SiF_6, F g/l	Bath composition: CrO_3, g/l	Ratio, CrO_3/F	Cathode current density, amp/sq ft	Cathode current efficiency, pct	Analysis, ppm[c]: O_2	Analysis, ppm[c]: N_2
39	4	6.1		300	49	1180	38	165	<50
41	6	5.4		300	56	1110	34	135	<30
43	8	5.3		300	57	1050	37	155	<50
45	11.5	5.3		280	53	1010	34	160	<50
47	3	6.5		300	46	980	33		
49[b]	6	5.3		300	57	880	31		20
51	3		5.2	300	57	1010	40	270	10
53[c]	12		5.3	300	57	1010	41		
55[b]	6		5.3	300	57	885	43		
57F1[d]	1		3.0	300	100	1035	32		
59F1	1		4.0	300	75	1020	39		
61F1	1		5.3	300	57	1025	42		
63F1	1		6.0	300	50	1040	40		
65F1	1		7.0	300	43	1035	29		
66F2[e]	1		8.7	500	57	1065	35		
69F2	1		5.3	300	57	1065	42		
71F2	1		1.8	100	57	1060	27		
73	1		5.3	400	75	1195	34		
75	1		5.3	200	38	1165	33	335	<10

[b] large-scale run
[c] recrystallized technical grade chromic acid
[d] F1 - data used in Fig. 1
[e] F2 - date used in Fig. 2

The electrolyte was prepared by dissolving reagent grade chromium trioxide in distilled water and adding the fluoride ion as hydrofluoric acid or the silicofluoride ion as hydrofluosilicic acid. No temperature control was necessary because heat generated during electrolysis was sufficient to maintain the bath temperature at the boiling point of 103°C.

The sulfonated polystyrene cation-exchange resin, which was successfully used for purifying sulfate electrolytes, decomposed in contact with solutions containing either the fluoride or silicofluoride ions. The sulfate ions thus introduced into the electrolyte caused a marked decrease in current efficiency[18, 19]. Low current efficiencies also resulted when technical-grade chromic acid was used. Technical-grade acid purified by recrystallization before use yielded results comparable to those attained with a reagent-grade material.

Experimental Results and Discussion

It was found that after a deposition interval of 4 to 5 hr, dendrites began to form and current efficiency was reduced considerably. Replacement of the cathode with a new one at this point eliminated dendrite growth and produced an adherent deposit about 1/16 in. thick. When more than one cathode was used in an experiment, the concentration and level of electrolyte were adjusted to their original conditions before inserting the new cathode.

Table II illustrates the important function of anion catalysts on current efficiency. Whereas the sulfate electrolyte produced maximum current efficiencies of 12 to 13 pct, efficiency values ranging from 33 to 37 pct were obtained for fluoride baths, and 40 to 43 pct for silicofluoride baths. In this respect, the present investigation is in general agreement with the work of Bilfinger[20].

The data appearing in Table II for silicofluoride baths indicate that at constant bath compositions the use of current densities lower than 850 and higher than 1100 amp per sq ft resulted in a slight decrease in current efficiency. These data are also depicted in Figs. 5 and 6. Fig. 5 graphically shows the relationship between cathode efficiency and fluoride concentration. This plot indicates that the optimum current efficiency was obtained for CrO_3 to F ratios between 56 and 59. Fig. 6 illustrates the effect of varying the chromic acid concentration on current efficiency while maintaining a constant CrO_3 to F ratio of 57. The optimum current efficiency was attained at a chromic acid concentration of 300 g per liter. On the basis of this work, the optimum conditions were established for silicofluoride electrolytes at the following levels:

Bath composition	300 g/l chromic acid 5.3 g/l fluoride ion
Cathode current density	850 to 1100 amp/sq ft
Operating temperature	103°C.

This work is in general agreement with the results on fluoride electrolytes reported in Australia by Ryan[22].

The oxygen content of deposits from fluoride and silicofluoride baths was on the order of 150 to 300 ppm and was, therefore, slightly higher than for metal obtained from sulfate baths. Regardless of the bath type used, no essential difference was noted in the nitrogen content of the deposited chromium. Metallic impurities did not vary in the different electrolytes except for tin which was found in the metal from both fluoride baths in amounts up to 100 ppm. This tin contamination resulted from attack on the 10% tin-lead alloy anode.

Physical Studies

In the preparation of ingots for physical testing, the deposits were first crushed, compressed into 1 x 1 in. bars 10-in. long, and sintered in purified hydrogen at 1200°C to increase their mechanical strength. These bars were consumable arc-melted into 2-in. diam ingots.

The ingots, used to prepare wire specimens for tensile tests, were scalped, sheathed in stainless steel, and swaged at 1100°C to approx 1-in. diam. By repeated swaging and recrystallization, the rods were reduced to ¼-in. diam. To obtain ⅛-in. diam test wire the rods were drawn through tungsten-carbide dies at 350°C. This wire could not be bent at this point and only after a thin surface layer had been removed by electroylic etching could the specimen be reverse-bent by hand repeatedly without fracture. The remarkable increase in wire ductility after etching was discovered at this laboratory[6,23] and by other investigators[23].

Due to the opportunities for atmospheric contamination during the process of preparing the specimens, the ingots as well as the specimens were analyzed for their nitrogen content. Tensile tests

Table III. Results of Tensile Tests of Wire at 25°C

Specimen No.	Ingot analysis, ppm O	H	N	Wire analysis, ppm N	UTS 1000 psi	Elongation, pct in 1-in.
W-88	245	7	<30	30	57	26
W-65	245	7	<30	40	69	10
W-82	245	7	<30	40	69	16
				50	68	16
SA-16, 284	40	2	<30	220	77	0
				—	74	0
SA-17, 286	40	2	<30	210	72	0
				—	70	0

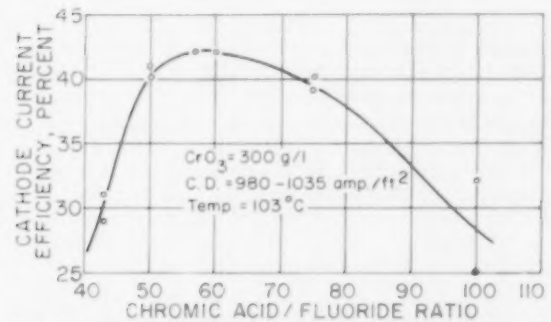

Fig. 5—Effect of fluoride concentration on current efficiency in silicofluoride baths.

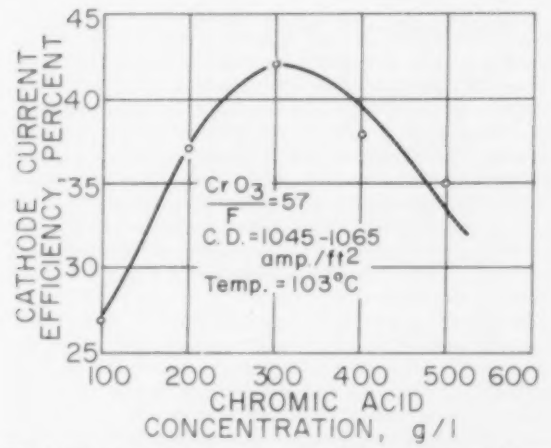

Fig. 6—Effect of chromic acid concentration on current efficiency in silicofluoride baths.

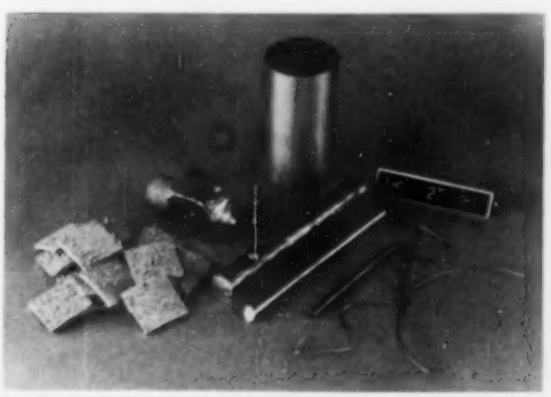

Fig. 7—Stages employed in preparing wire from electrodeposited chromium.

representing specimens prepared from two ingot melts are given in Table III. These results indicate that chromium containing 50 ppm or less N was ductile, while metal containing 200 ppm N had no ductility. The tests reveal that very small increases in the nitrogen adversely affect the ductility of wire, and point out the importance of preparing and maintaining low-nitrogen metal.

The chromium deposited from fluoride or silicofluoride baths, while not subjected to extensive physical testing, should compare favorably with metal obtained from the sulfate baths. No large differences were noted in the nitrogen content of electrodeposits from the different types of baths. In addition, the slightly higher oxygen content of fluoride or silicofluoride deposits should have little effect on ductility, since numerous investigators[1,6,11,22] have indicated that small amounts of oxygen do not cause brittleness in chromium metal.

Fig. 7 represents the stages employed in preparing chromium wires; it shows the electrodeposit, a section of partly-melted consumable electrode, an ingot section, swaged rods, and drawn-etched wires.

Conclusion

Kilogram quantities of high-purity, ductile chromium were prepared by electrodeposition from purified chromic-acid solutions at elevated temperatures. Although the fluoride-type baths have a better throwing power and a higher efficiency than baths containing the sulfate additive, the metal deposited from sulfate electrolytes appears to be of better quality because it contains slightly less oxygen and tin than metal obtained from the fluoride-type electrolytes.

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PRODUCTION OF UF_4 FROM REFINERY SCRAP MATERIALS

Under the auspices of the AEC the National Lead Co. has endeavored to develop a method of producing uranium from scrap. The authors compare the relative difficulty and cost of the prior operations to their method in addition to a detailed discussion of the present mode of activity.

by E. O. Rutenkroger, T. J. Collopy, J. H. Cavendish and M. A. De Sesa

At the AEC Feed Materials Production Center, which is operated by the National Lead Company at Fernald, Ohio, uranium concentrates are refined and converted to uranium metal. The metal is fabricated into fuel elements for plutonium production reactors. The various processes used in uranium metal production are familiar technology by now[1], but are outlined schematically in Figs. 1 to 3 in order to show the points of generation of scrap material and the present process for recycle of this scrap material to the production stream.

As illustrated in Fig. 1, incoming ore concentrates are sampled and then transferred to digestion tanks for dissolution in concentrated nitric acid. Two types of scrap material are commonly added to each digestion batch, U_3O_8 or black oxide and calcined UAP. Each of these materials is generated in the Metals Recovery Plant. Approximately 50 pct of the scrap uranium tonnage received at the Metals Recovery Plant is merely burned to black oxide and sent to the Refinery. Those scrap materials low in uranium content or high in fluoride are converted by a hydrometallurgical process to uranyl ammonium phosphate (UAP)[2]. The UAP is calcined to produce a uranium pyrophosphate which is then recycled to digestion. Massive metal scrap is dissolved separately in nitric acid and then both nitric acid streams are combined in hold tanks. The crude uranyl nitrate solution is purified by solvent extraction with tri-n-butyl phosphate to yield a pure uranyl nitrate solution.

In Fig. 2, the processes used to produce UF_4 green salt, are illustrated. The uranyl nitrate solution is evaporated and boiled down to the approximate composition of uranyl nitrate hexahydrate (UNH). The UNH is fed to denitration pots where it is converted to UO_3 orange oxide. Orange oxide is reduced with hydrogen in fluid bed reactors and then hydrofluorinated in screw reactors to produce UF_4 green salt. Any off-specification UO_3 which can not be blended or otherwise consumed is returned to digestion. Also any off-specification UF_4 which can not be consumed or sold is processed in the Metals Recovery Plant to UAP.

As shown in Fig. 3, the UF_4 produced by the chemical processes just illustrated is blended with magnesium and reduced to uranium metal by a thermite reaction. The magnesium fluoride scrap from this step is a major source of low-grade feed material to the Metals Recovery Plant. The cleaned massive piece of uranium metal, known as a derby, is remelted with various remeltable scrap and cast into an ingot. Subsequently, the graphite crucible is cleaned of an adhering thin layer of metal by a "burn-out" procedure to produce a black oxide with a high fluoride content. All contaminated chips and sawdust of uranium metal produced during machining are sent to the Metals Recovery Plant for calcination to U_3O_8.

This review of the processes at the Fernald plant has demonstrated how each available scrap material is recycled. As has been shown, much of this material is returned to the head end of the plant for reprocessing through the entire circuit. This is an expensive recovery process and the quantities which can be processed in the Refinery are restricted due to the phosphate content of UAP and the high concentration of fluoride and carbon in the black oxides. In this paper, the laboratory work leading to a process for the direct preparation of green salt from scrap materials will be presented. Also described is the Pilot-scale work performed to substantiate the laboratory results and to provide sufficient quantities of UF_4 for metal reduction evaluations. The results of small-scale metal reduction tests are summarized.

Experimental

A process for the preparation of green salt from uranium ores and concentrates was reported in 1958 from the AEC Raw Materials Development Laboratory at Winchester, Mass.[3-6]. Commencing with a sulfuric acid solution of uranium prepared from either ore or concentrate, the uranium is extracted into a kerosene solution of an amine. The uranium is recovered in a concentrated and purified form by stripping the amine with hydrochloric acid. Ura-

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nium tetrafluoride is precipitated from the hydrochloric acid strip solution by a unique process. Hydrofluoric acid and cupric sulfate are added to the pregnant strip solution and UF_4 precipitated by bubbling SO_2 through the hot solution. Reduction of hexavalent uranium to tetravalent uranium is accomplished by the catalytic action of the cuprous chloride ion which is continuously regenerated from the cupric chloride ion by the addition of sulfur dioxide to the solution during the precipitation step. The following equations represent the pertinent reactions:

$$2\,CuCl_4^{=} + SO_2 + 2H_2O \rightarrow 2\,CuCl_2^{=} + SO_4^{=} + 4H^+ + 4Cl^-$$
$$2\,CuCl_2^{=} + 4HF + UO_2^{++} + 4Cl^- \rightarrow UF_4\downarrow + 2\,CuCl_4^{=} + 2\,H_2O$$
$$UO_2^{++} + SO_2 + 4HF \rightarrow UF_4\downarrow + SO_4^{=} + 4H^+$$

The uranium tetrafluoride product which is produced by this procedure is a high density material which is easily filtered. Final product purity is a function of both the purity of the starting solution and the particular degree of decontamination obtained during precipitation for each individual impurity. Material meeting metal-grade or other specifications can be produced by control of these two variables. The foregoing process is currently being adapted at the National Lead Company of Ohio for the recovery of uranium as uranium tetrafluoride from low-grade and high-grade scrap.

Preparation of UF_4 from low-grade scrap

A flowsheet which was developed in the laboratory for the treatment of low-grade scrap is presented in Fig. 4. The details of the laboratory work which led to the development of this flowsheet have been reported in NLCO-788[6]. Metals Recovery Plant prepared feed—6-10 pct uranium—is leached with sulfuric acid using manganese dioxide as an oxidant to prepare a feed solution containing 30 gpl U and 2N H_2SO_4. The uranium is extracted from this solution into a 5 volume pct solution of Rohm and Haas amine LA-1 (formerly designated as 9D-178) in kerosene containing 2.5 volume pct primary decyl alcohol. After three stages of mixer-settler extraction at an organic to aqueous ratio of 7.5 to 1, the raffinate contains less than 0.01 gpl U and 1N H_2SO_4. This raffinate is neutralized and discarded. Note that about half the sulfuric acid entering in the feed solution is consumed in converting the free amine to the bisulfate form.

The loaded organic is stripped with 3N HCl in one stage at an organic to aqueous ratio of 20 to 1 —only one stage of stripping is used. The nature of the stripping equilibrium curve is such that even when using four stripping stages the minimum uranium concentration of the stripped organic is about 0.03 gpl. An organic to aqueous ratio of 14:1 in carbonate conversion, would result in an aqueous stream assaying around 0.4 gpl uranium, which could not be discarded. Since 90 to 95 pct of the uranium is stripped in the first stage, only one stage of stripping is used to produce as concentrated a strip solution as possible—desirable for the precipitation step. The uranium remaining in the organic phase is recovered by carbonate stripping. [5]The amine solvent now in the free amine form is recycled. The carbonate wash solution is neutralized with an acidic filtrate from UF_4 precipitation and the uranium recovered by precipitation with ammonia.

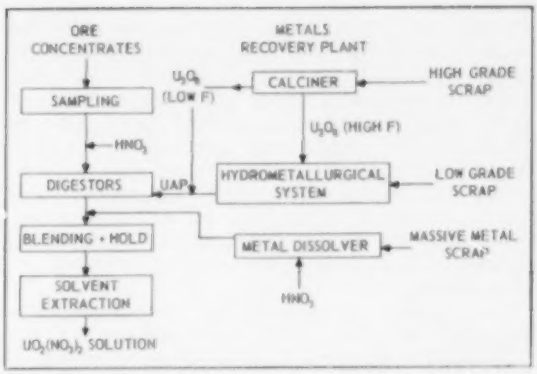

Fig. 1—Preparation of refined uranyl nitrate solution from ore concentrates and recycled scrap material.

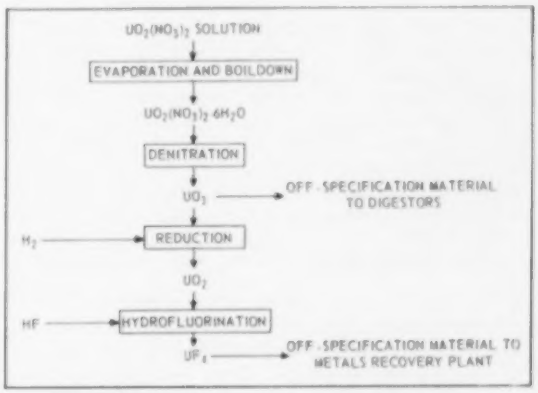

Fig. 2—Conversion of uranyl nitrate solution to UF_4.

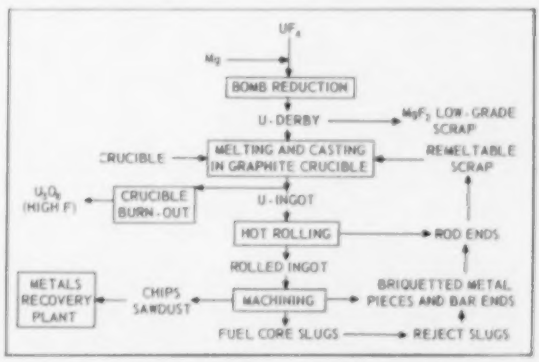

Fig. 3—Metal production and finishing operations.

Copper (5.6 gpl as $CuSO_4 \cdot 5H_2O$ and HF (15 gpl in excess) as a 50 pct solution are added to the hydrochloric acid strip solution. The uranium is precipitated as $UF_4 \cdot 3/4\,H_2O$ by heating the solution to 90° to 95°C and bubbling SO_2 gas into the solution. The filtrate from the precipitation step may be neutralized to pH 2.0 with NH_4OH and the copper recovered by plating onto scrap iron. The copper plates onto the iron in a spongy form which is easily removed by a water spray.

Preparation of UF_4 from high-grade scrap

The uranium ammonium phosphate (UAP) product and the various high-grade black oxides from the Metals Recovery Plant were evaluated as feeds

for a process involving hydrochloric acid dissolution followed by UF_4 precipitation according to the Winlo* process. Initial scoping tests were conducted using uncalcined UAP and calcined UAP—both samples were taken from the Metals Recovery Plant production operation. The UF_4 produced from uncalcined UAP was found to contain 1 to 2 pct NH_3 and to have a relatively low tap density (2.5 g per cu cm). Because of the low density and high ammonia content of this UF_4, all subsequent tests were conducted with calcined (1200°F) UAP which apparently is a mixture of uranyl and uranous pyrophosphates.

Two variables were examined in the precipitation of UF_4 from hydrochloric acid solutions of calcined UAP, uranium concentration and excess hydrochloric acid concentration.

A series of HCl dissolutions was conducted to produce solutions assaying approximately 100, 200, and 300 gpl U. The dissolutions were carried out employing the stoichiometric amount of HCl (based on the reaction, $(UO_2)_3P_2O_7 + 4\ HCl + H_2O \rightarrow 2\ UO_2Cl_2 + 2H_3PO_4$) plus a 1N excess. In order to obtain complete dissolution, it was necessary to use elevated temperatures (80° to 90°C) and HNO_3 as an oxidant for the reduced uranium portion of the calcined UAP.

Following dissolution of the calcined UAP, the solutions were filtered to remove the insoluble filter aid which was present in the UAP cake. Green salt was then precipitated from the solutions by adding 22 gpl $CuSO_4 \cdot 5\ H_2O$ and a 15 gpl excess of HF (added as a 50 pct solution) and bubbling SO_2 gas into the solutions. All of the precipitations were conducted at 90° to 95°C. The precipitated UF_4 was oven-dried at 110°C and was analyzed wet-chemically and spectrochemically. The results of these tests are shown in Table I.

Table I. Effect of Uranium Concentration on UF_4 Purity From Leach Solutions of Calcined UAP

	Calcined UAP Feed	Test No. 1	2	3
		HCl Leach Solution		
U (g/l)		95	176	293
PO_4 (g/l)		38	67	—
H+(N)*		1.9	2.4	3.4
		UF_4 Product**		
Wet Chemical				
AOI (pct)		0.02	0.02	0.04
U (pct)	61.7	72.98	73.22	73.70
U^{+4} (pct)		72.40	72.58	72.77
UF_4 (pct)		96.33	95.53	95.99
H_2O (pct)		2.90	3.62	2.77
F (pct)		23.00	23.04	23.24
PO_4 (pct)	26.5	0.13	0.09	0.08
Tap Density (g/cc)		2.91	2.91†	3.26
Spectrochemical (ppm)††				
Al	400	15	20	15
Ba	—	30	—	45
Bi	<20	<1	<1	<1
Ca	—	180	—	150
Cd	<200	<0.2	<0.2	<0.2
Cr	<100	<10	<10	<10
Cu	300	35	25	6
Fe	3000	50	80	99
Li	—	<2.5	—	<2.5
Mg	1600	>100	>100	>100
Mn	650	<4	<4	<4
Mo	<10	<6	<6	<6
Na	—	75	—	<200
Ni	<20	<10	<10	<10
P	>60,000	<20	<20	<20
Pb	80	2	2	<1
Sn	150	<1	<1	<1
Sr	<20	<10	—	<10
V	<100	<40	<40	<40
Zn	<200	<20	<20	<20

* As titrated with NaOH to pH 6.05.
** Oven-dried at 110°C.
† Grinding of sample in mortar increased the tap density to 3.51 g/cc.
†† On uranium basis.
— Not determined.

As may be seen, the chemical purity of the product UF_4 is good except for calcium, magnesium, sodium, and possibly phosphorus. It is considered probable that good decontamination of these materials would be obtained during metal reduction. The tap density of the UF_4 varied from 2.9 to 3.2 g per cu cm. Grinding of the sample produced in the 200 gpl test increased the tap density from 2.9 to 3.5 g per cu cm.

The initial uranium concentration of the leach solution appeared to have little effect on the impurity level with the exception of iron and sodium. These two impurities seemed to increase as the starting uranium concentration was increased. The precipitation times for the runs at 100, 200, and 300 gpl were 1.5, 3.0 and ~ 7 hours, respectively. This indicates a reduction in SO_2 efficiency at the 300 gpl level (probably due to decreased solubility of SO_2 in H_3PO_4). On the basis of these tests, a uranium concentration in the range of 200 gpl was chosen for further tests.

Since the chloride concentration of the feed solution is reportedly one of the rate-controlling factors in the precipitation of UF_4 by the Winlo process*, a brief laboratory investigation was carried out to study the effect of HCl concentration on the precipitation. A series of dissolutions and aqueous UF_4 precipitations were conducted in which the HCl excess used in dissolution of the uranium pyrophosphate was 0.0, 1.0, and 2.0N. The dissolutions and precipitations were conducted in the same manner as those described in the previous section and at the 200 gpl U level. The results of these tests indicated that the HCl excess (in the range studied) has no significant effect on the quality of the resulting UF_4. However, the time required for complete precipitation increased slightly as the HCl excess was increased. The times for the runs at 0.0, 1.0 and 2.0N excess were 2.4, 3.0 and ~ 3.5 hours. Previous work has shown this same effect to be much more pronounced in an H_2SO_4—HCl system than in a starting system of HCl alone. It appears that the H_3PO_4—HCl system is similar to the H_2SO_4—HCl system in this respect. On the basis of these tests a 1.0N HCl excess was chosen for further tests.

The various high-grade black oxide types of scrap resulting from the burning of chips, turnings, sludges, etc., have been investigated in the laboratory to determine their amenability to an HCl dissolution-aqueous UF_4 precipitation process. The purification which could be obtained by this technique was of primary interest. Samples used for laboratory testing were composite lot samples which were representative of the production of three months' operation during the period Jan. 1, 1959 to Mar. 31, 1959. Samples of the following types were investigated: crucible burnout black oxide, oxidation furnace ash, graphite furnace ash, box furnace ash, and alloyed black oxide.

The laboratory tests were conducted by dissolution of the black oxides in HCl (using HNO_3 as an oxidant) to produce solutions for precipitation —approximately 200 gpl uranium and 1N excess

* The name given to the aqueous precipitation process for the precipitation of UF_4 developed at the Winchester Laboratory and reduced to practice at the National Lead Co. of Ohio.

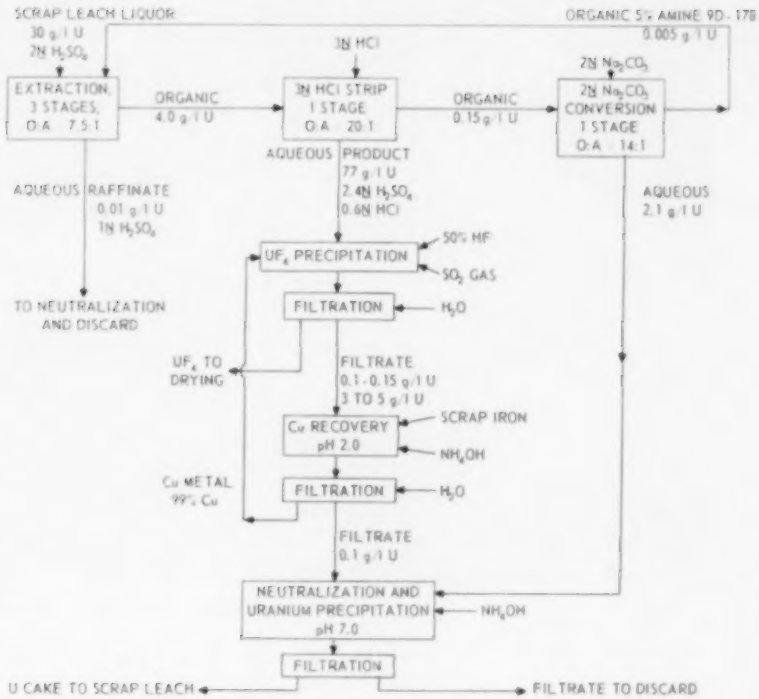

Fig. 4—Flowsheet for amine extraction—aqueous UF_4 precipitation process for scrap.

HCl. The insolubles were removed prior to UF_4 precipitation. The clarified solutions were precipitated by adding 22 gpl $CuSO_4 \cdot 5\ H_2O$ and a 15 gpl excess of HF, and bubbling SO_2 into the solution. The precipitations were all conducted at 90° to 95°C and required 2 to 2.5 hr for complete precipitation. Feed used in each test was analyzed spectrochemically, and the resulting UF_4 was analyzed both wet-chemically and spectrochemically. The results of the tests indicated that all of the samples tested produced high-grade UF_4 which was low in impurities. The UF_4 produced from all of the materials tested had an average assay of 0.19 pct AOI, 0.44 pct UO_2F_2, 96.03 pct UF_4, and 3.35 pct H_2O. It appears that dehydration to remove the water content would produce a UF_4 which would meet metal-grade specifications in all respects.

Pilot-plant test of the process

The possibility of producing green salt directly from UAP and high-grade scrap material looked so promising on the basis of laboratory tests, that a series of pilot-scale tests was made to further evaluate the process and to produce enough green salt for metal evaluation tests.

A series of dissolution and precipitation tests was conducted in the Pilot Plant semi-works (200 gal) unit which is shown in Fig. 5. The feed used for these runs was prepared by blending various black oxides and calcined UAP obtained from the Metals Recovery Plant production. The blend used and the uranium content of the feed materials are shown in Table II.

A total of 21 digestions were made. The digestions were conducted by adding the feeds to a hydrochloric acid solution in proportions calculated to yield 140 gallons of digest filtrate containing 200 gpl uranium and a 1N HCl excess. A small amount (4 gallons) of 60 pct HNO_3 was added to each batch as an oxidant. Digest batches were all agitated for 2 to 3 hr at 85° to 95°C (185° to 203°F) before filtering. Uranium recovery efficiency was checked on some of the digest batches and was found to average 99.7 pct. The analyses of the digest filtrates from the 21 digestions averaged 197 gpl U, 48 gpl PO_4, and 1.9N in free acid.

Table II. Feed Samples for Pilot Plant Tests

Material	Pct of Blend (U basis)	U pct	PO_4 pct
Calcined UAP	50	57.3	28.9
Graphite Furnace Ash	6	86.2	—
Oxidation Furnace Ash	20	84.6	—
Box Furnace Ash	7	84.9	—
Crucible Burnout U_3O_8	17	83.7	—

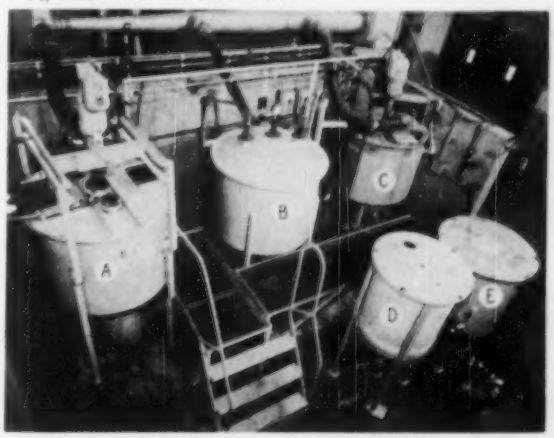

Fig. 5—The 200-gal pilot plant semi-works installation. A is a 200-gal rubber-lined tank, B a 200-gal rubber-lined vacuum receiver, C a precipitator (200 gal Hastelloy B tank), D a polyethylene storage tank, and E a rubber-lined storage tank.

Hydrated uranium tetrafluoride ($UF_4 \cdot 3/4\ H_2O$) was precipitated from the filtrate of each of the digestion batches by adding 22 gpl $CuSO_4 \cdot 5\ H_2O$ and a 15 gpl excess of HF (~ 30 pct HF from the Green Salt Plant off-gas system) and then bubbling SO_2 into the solution. The precipitations were each conducted at 85° to 95°C (185° to 203°F)—4 to 6 hr being required for complete precipitation. Upon completion of the precipitation, the slurries were filtered and the filtrates were analyzed for uranium and fluoride. The analyses averaged 0.16 gpl uranium and 16 gpl fluoride, for a uranium recovery in excess of 99.9 pct. The filter cake was dried in a HoloFlyte conveyor. Approximately 300 pounds of $UF_4 \cdot 3/4\ H_2O$ were produced from each of the 21 runs, for a total production of 6300 lb. Samples of the product from each of the runs were analyzed both wet-chemically and spectrochemically.

The average analyses are shown below:

Wet Chemical		Spectrochemical (ppm on U basis)			
AOI (pct)	0.03	Al	>100	Mn	8
UF_4 (pct)	94.39	Bi	<1	Mo	<6
U$^+$, (pct)	71.54	Ca	>200	Na	>200
F (pct)	23.38	Cd	<0.2	Ni	<10
H_2O (pct)	3.71	Cr	10	P	>200
Tap Density (g/cc)	2.91	Cu	18	Pb	2
		Fe	181	Sn	<1
		K	>200	V	<40
		Li	<5	Zn	<20
		Mg	>200		

The wet chemical analyses indicate a total impurity content of almost 2 pct. It is assumed that most of the impurity content is due to alkali and alkaline earth fluorides which would be largely removed in reduction of the green salt to metal.

One dehydration run was made to produce some dehydrated UF_4 for metal reduction tests. Approximately 260 lb of $UF_4 \cdot 3/4\ H_2O$ was dehydrated in this run. The run was conducted in a Monel reactor containing stacked trays which had cutouts to allow for gas flow. The reactor was equipped with two small openings, a gas inlet and a gas outlet. Each of the trays was loaded to about one inch thickness with $UF_4 \cdot 3/4\ H_2O$. Dehydration was accomplished by placing the reactor in a Rockwell furnace and slowly raising the temperature to 820°F. When no more steam was visible at the off-gas exit line, the reactor was pulled from the furnace and sparged with air for 10 min. The reactor was allowed to cool before the trays were removed from it. The UF_4 produced has the following assay: UF_4 pct 98.17, AOI pct 0.14, UO_2F_2 pct 1.2, U$^+$, pct 74.54, F pct 23.72, H_2O pct 0.13 and a tap density (g per cc) 3.23. As can be seen, excellent dehydration was accomplished with the formation of very little AOI. The UO_2F_2 content is also in the desirable range of 1 to 2 pct.

The technique of dehydrating Winlo green salt in its own atmosphere was devised on a laboratory scale at the Raw Materials Development Laboratory[7]. Dehydration in a closed vessel tends to repress the hydrolysis of UF_4 to UO_2; and the final air sparge is used to produce some UO_2F_2 which is desirable during the reduction to metal process.

Small-scale bomb reduction evaluation of Winlo green salt

Some of the material generated in the pilot plant tests was used in small-scale bomb reduction tests to determine whether uranium metal of acceptable quality could be made by this process. In this technique approximately 33 lbs of green salt are reduced in each charge. The results of this investigation may be summarized as follows:

1. Blends of 25 w/o hydrated Winlo green salt with 75 w/o production green salt (hydrofluorinated UO_2) having an average blend composition of 95.5 per cent UF_4 produced crude yields comparable with those obtained when only production green salt was used. Preheat times for the blended green salt were 50 to 66 pct longer.
2. A blend of 25 w/o dehydrated Winlo green salt (less than 0.2 w/o H_2O) and 75 w/o production green salt produced crude yields that were comparable with those obtained with production green salt, preheat time being increased only slightly in comparison with production green salt.
3. Hydrated Winlo green salt did not reduce to metal. When unblended Winlo green salt was dehydrated to less than 0.2 w/o H_2O, a very low crude derby yield (50 pct) was obtained. But when it was dehydrated and then ground to have a distribution of particles 20 μ and smaller (comparable with production green salt), it produced crude yields that were comparable with those obtained with production green salt. Preheat times being only slightly increased in comparison with production green salt. The difficulties encountered in attempting to reduce Winlo green salt without prior blending with production green salt or without prior grinding seem to be due to segregation of the UF_4 and Mg in the bomb prior to reduction.
4. The metal quality of the uranium produced from the Winlo green salt was comparable with that produced from the production green salt.

Summary

On the basis of laboratory and pilot-scale tests it has been shown that both low-grade and high-grade uranium scrap materials can be converted to UF_4 in the Winlo process. Low-grade scrap materials should be upgraded in quality by amine solvent extraction or conversion to UAP. Many high-grade scrap materials can be treated directly by a HCl dissolution—Winlo precipitation process.

Winlo green salt can be blended in the hydrated form with production green salt prior to reduction to metal. Winlo green salt alone can be reduced to metal if it is dehydrated and ground.

Processing of scrap material directly to green salt by the Winlo process will reduce the over-all costs of metal production and relieve the load on the chemical processing plants by eliminating recycle of scrap material.

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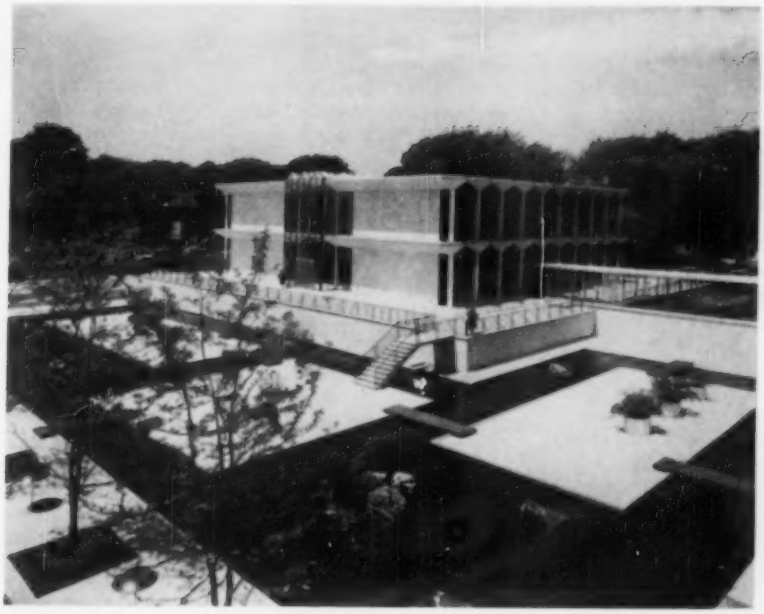


McGregor Center, Wayne State University

Refractory Metals à la Detroit

Ultramodern McGregor Center on the Wayne State University Campus, Detroit, was filled to near capacity by refractory metallurgists May 25-26. Reason: a symposium on Refractory Metals and Alloys, sponsored by the Institute of Metals Division and the Detroit section of AIME.

Julius J. Harwood, Chairman of the IMD Refractory Metals Committee, greeted the 400 metallurgists with an introduction to the *state of the art* of refractory metals—a subject which was thoroughly explored in the course of the two-day meeting. At the same time, he announced that there would be no recorded discussion, but that written discussion was invited for the meeting's proceedings volume.

Leading off the first session was William Rostoker of Armour Research Foundation. Dr. Rostoker spoke on *Phase Equilibria in Refractory Metal Systems*. He noted that a phase equilibria program should prescribe limited effort to maximum utilization. Included in the paper was a method for pinpointing troubles encountered in the use of various compounds at elevated temperatures.

Mechanical Behavior of Refractory Metals and Alloys delivered by James Bechtold, Westinghouse Electric Corp., was an assimilation of the literature on the mechanical behavior of refractory metals. Co-authors were E. Wessel and L. France, also of Westinghouse. The paper elicited much pencil pushing and even clicks of several cameras in the audience when the diagrams were flashed on the screen.

Strengthening of Refractory Alloys by W. H. Chang, Flight Propulsion Laboratory dept., General Electric Co., represented the third of the basic papers which served as an introduction to the subject.

Now there was a break for lunch, and the 200 who were lucky enough to get luncheon tickets settled down to some socializing and victuals. Later, it was back to the airy, acoustically-excellent auditorium for resumption of activities. But before the technical tempest was continued, AIME President, Joseph L. Gillson was introduced. Dr. Gillson welcomed those present and commented on the merits of AIME membership.

Next to operate the light pointer from the lectern was N. E. Promisel, Director, Materials div., Bureau of Naval Weapons, Dept. of the Navy. "Contrary to custom," Mr. Promisel explained, "I will begin by showing a slide." In just that light and informative vein did he describe AGARD, the Advisory Group for Aeronautical Research Development. Mr. Promisel pointed out that the Group functions under the North Atlantic Treaty Organization (NATO), and that two high-priority materials problems faced by NATO countries concern refractory metals and graphite.

More ponderous after-luncheon fare was served up in a paper by J. W. Semmel, Jr., Flight Propulsion Laboratory dept., General Electric Co. in *Oxidation Behavior of Refractory Metals and Alloys*, Mr. Semmel noted that improved oxidation-resistance has been obtained in potentially-useful alloys of all the base metals, except molybdenum. However, the oxidation resistance of alloys is still below that required for general purposes. Chromium alloys appear most promising, in view of the progress made in decreasing nitrogen absorption.

Edward Fox and J. A. McGurty, Aircraft Nuclear

Propulsion dept., General Eletcric Co., authored the fifth paper on the program, entitled *Chromium and its Alloys*. Another treatment of the topic was given by Robert Widmer, now technical director, New England Materials Laboratory. Toru Yukawa, formerly a research asst., Massachusetts Institute of Technology and N. J. Grant, professor of metallurgy, MIT, were the co-authors of *Oxidation Behavior of Chromium-Base Alloys at Temperatures Between 1800° and 2200°F*.

Next to be heard was Fred J. Anders, Jr., E.I. du Pont de Nemours & Co. Inc. *Metallurgy of Columbium and Its Alloys* was the subject of his talk. *The Present and Future of Columbium Alloys* by R. G. Frank, Flight Propulsion Laboratory dept., General Electric Co., described making usable hardware from columbium. There was time for a little discussion following the second of the columbium papers and both authors were questioned.

Paper No. 9 was *Molybdenum and Its Alloys* by M. Semchyshen and R. Q. Barr, Research Laboratory, Climax Molybdenum Co. of Michigan. Mr. Semchyshen was also Chairman of the Detroit section, AIME Host Committee, and to him should go accolades for diligence in seeing that the authors did not violate their limits too fragrantly.

Thus ended the first day of technical papers. Registrants soon retired to the social hour which was sponsored by the Detroit section, and then went on to other social pursuits.

Second day

Tungsten and Tungsten Base Alloys led off the final day's activities. The paper was authored by R. Atkinson and R. C. Koo, Research Laboratories, Westinghouse Lamp div.; Mr. Atkinson delivered the paper. Sigmund Weissmann, Rutgers University next delivered a discussion based on his X-ray studies of tungsten.

Tantalum and Tantalum Alloys was the subject of a paper by A. B. Michael, Fansteel Metallurgical Corp. It was both an historical analysis and a review of tantalum and its alloys.

Now it was time out for a coffee break, and when the majority could again be corraled in the auditorium, R. I. Jaffee of Battelle Memorial Institute delivered a paper on *Rhenium and the Refractory Platinum-Group Metals*. Co-authors were D. J. Maykuth and R. W. Douglass of Battelle. These metals are relatively easy to produce in high-purity form, explained Dr. Jaffee, but they pose difficulties in fabrication, the difficulty increasing in the following order: rhodium, iridium, rhenium, ruthenium, and osmium. Low-temperature ductility problems are encountered, particularly in iridium, ruthenium, and osmium. In fact, it was pointed out, osmium has yet to be successfully fabricated. As to oxidation resistance, it ranges from poor for rhenium, osmium, and ruthenium, to the best of any known metal for rhodium. In regard to alloying effects of rhenium in group VI, metals (Cr, Mo, W), investigation indicates that the refractory platinum-group metals possess many, but not all, of these characteristics.

The last paper presented before the luncheon break was *High Temperature (2000°F) Strength Properties of Refractory Metals and Alloys*. R. W. Hall, Lewis Research Center, National Aeronautics and Space Administration, delivered the paper. Co-workers, P. F. Kikora and G. Mervin Ault helped with the research and paper.

The last session

Back to work after lunch, but with some lighter after-lunch fare. Chairman Harwood read his translation from the French of Robert Syre's paper, *Survey of European Development in Refractory Metals*. Mr. Syre, of the French firm, Pechiney, was unable to attend.

Fabrication and *Processing of Refractory Metals* was next on the agenda, with C. P. Mueller, Refractomet div., Universal-Cyclops Steel Corp., doing the honors. Mr. Mueller described work on two systems: molybdenum-titanium and molybdenum-columbium. His slides on application of refractory metal sheet in air frames and other hardware provoked much picture snapping from the audience. And his predictions on the revolutionary era which refractory metals are approaching stimulated no little amount of interest. Equipment developments, Mr. Mueller says will make this possible. And the machinery and material on which he bases this prediction is, in part, Cyclops' own InFab (Inert Fabrication) facility. This inert argon atmosphere room, impactor, rolling mill, and furnaces were described in the August issue of Journal of Metals, p. 636.

The last paper on the schedule was *Protective Coatings for High Temperature Environments*. E. D. Sayre, Flight Propulsion Laboratory Dept., General Electric Co., representing his co-authors, L. Luft and J. Graham, read the paper.

Mr. Sayre outlined the criteria for coatings needed to protect refractory metals against oxygen and nitrogen diffusion. Also included in the well-organized paper were some results on development of protective coatings for molybdenum and columbium used at 2500°F. The plots on the graphs precipitated much neck craning by the audience.

It was now 2:50 pm, and the meeting which had started out in arrears time-wise was now ahead of schedule. Thus, more time was allowed a choice panel of experts to consider refractory metal applications. Moderating the panel on the Comparative Status of Refractory Metals for Engineering Applications was W. J. Harris, Jr., executive director, Materials Advisory Board, National Academy of Sciences. Dr. Harris also is an AIME Director from the Institute of Metals Division. Sitting on the panel were E. Czarnecki, Boeing Aircraft Co.; G. M. Ault, Lewis Research Center, NASA; R. A. Perkins, Solid Rocket Plant, Aerojet-General Corp.; A. V. Levy, Aircraft div., Hughes Tool Co.; and P. Patriarca, Oak Ridge National Laboratory.

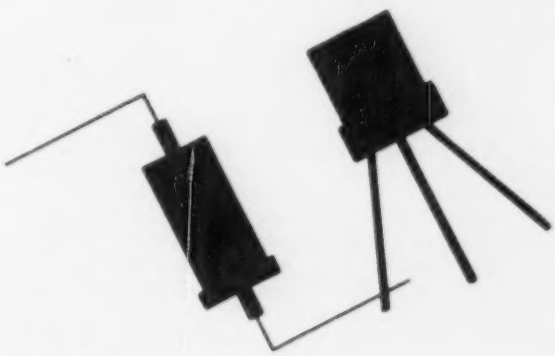
Mr. Czarnecki tackled the application of refractory metals from the standpoint of advanced weapon systems; Mr. Perkins on missiles; Mr. Levy on ramjets; and Mr. Patriarca on nuclear applications. Each of them explained the part played by refractory metals in his own space-age application. Then it was time for questions from the audience, and questions were forthcoming from the too-long silenced onlookers. The differing partialities of the panelists prompted a stimulating discussion of the relative merits and demerits of columbium, molybdenum, and tungsten for defense purposes.

Their tongues loosened and their interests satiated after two days jam-packed with of technical papers, the 400 metallurgists once again turned to the highways and byways . . . with perhaps a little more knowledge that could help them in their jobs.

RMS

BEANS, PILGRIMS, AND SEMICONDUCTORS

A report on the Technical Conference, "Metallurgy of Elemental and Compound Semiconductors", sponsored by IMD's Semiconductors Committee and the Boston section of AIME—August 29-31, 1960.



Traditional August hotel doldrums were shattered once again this year in Boston as over 540 metallurgists, chemists, and solid-state physicists converged on *Beantown* for The Metallurgical Society's Conference on the *Metallurgy of Elemental and Compound Semiconductors*. This second meeting in what is intended to become a series of annual conferences was held at the Statler-Hilton Hotel from August 29-31 under the joint sponsorship of the Institute of Metals Division's Semiconductors Committee and the Boston Section of AIME.

The program for this year's conclave, expanded to five sessions, featured a variety of subjects, including Esaki-Tunnel diodes, dendritic and vapor growth, and growth of materials other than those discussed in connection with the former techniques.

Welcoming registrants to the opening session on Monday morning, Conference Chairman David C. Jillson reported on the Semiconductors Committee's determination that its series of annual conferences should become the primary forum for discussing and reporting the latest advances in the metallurgy of semiconductor materials. He also reported on the tentative schedule of locations for the meetings in future years: 1961, Los Angeles; 1962, Philadelphia; 1963, Chicago; and 1964, Boston. Dr. Jillson stressed that geographical rotation had been planned as a means of bringing the conference to centers of semiconductor metallurgy throughout the country.

Registrants attending the fellowship dinner Monday evening heard an interesting description of *Building Businesses on Scientific Frontiers* as related by William Congleton, technical director of the American Research and Development Corp. in Boston. The speaker described the successes—and occasional failures—with which his company has been connected as a source of venture capital for new businesses. Of particular interest were Mr. Congleton's examples, almost all of them being drawn from companies based on technological developments, many of which were well known to the audience.

Besides the Chairman, D. C. Jillson, General Electric Co., those who contributed to the success of the conference in various connections included G. E. Brock (Publicity), International Business Machines; H. E. Bridgers (Program), Bell Telephone Laboratories; Pascal Levesque, Raytheon Manufacturing Co., H. I. Dixon, Metallurgical Products Co., and Samuel Valencia, Watertown Arsenal, (Membership Committee); and Prof. T. B. King (Local Arrangements and Registration), Massachusetts Institute of Technology.

The proceedings of the Conference will be edited by R. O. Grubel, General Electric Co., and published in mid-1961 as a volume in the Metallurgical Society Conference Series.

DCJ

Technical Sessions

by H. E. Bridgers

Judging from the packed attendance at each session, it is clear that the theme of the conference was timely and that the quality of the papers presented was excellent.

Dacey, of Bell Telephone Laboratories, gave an invited paper concerning the material requirements for Esaki diodes. This was approached from the point of view of the various figures of merit of the finished device. He discussed the maximum frequency cutoff, negative resistance, noise, switching voltage and current, and how these device requirements were met by the various materials at our disposal. Such materials as indium antimonide would give very satisfactory low-noise performance. Other materials like gallium arsenide would be advantageous for switching applications, allowing a larger fan-out in computer operations.

As a complimentary invited talk, Trumbore, of Bell Telephone Laboratories, discussed specific techniques to grow heavily-doped materials with distribution coefficients, or k's, greater than 1. He emphasized the need for closer control of growth parameters, lower pulling rates, and reduced temperature fluctuations. He discussed the role of crystallography on trapping impurities and the need for flat growth interfaces free of facets. He also discussed other techniques of growth, and in particular, the thermal gradient method and the solvent evaporation technique. Patel, of Raytheon, also discussed heavily-doped materials. The introduction and movement of dislocations in heavily-doped material was considered, and it was shown that there was a difference in the dynamic behavior of imperfections, depending on the doping impurity.

Skalski, of Radio Corp. of America, discussed the growth of gallium arsenide from dilute solutions. Particularly interesting was growth from gallium solution with its resultant low temperature. Growth from arsenic solution takes place at higher temperatures and much higher pressures. Seidel, of Radio Corp. of America, spoke on Hall measurements on tunnel-diode material. For higher doping levels he showed that phosphorus-doped material had higher mobility than comparably-doped arsenic materials. In the same connection, he discussed the role of the impurity-banding phenomena and questioned our knowledge of the dielectric constant and effective mass for these materials. He also discussed infrared absorption measurements and the use of this technique to obtain a ratio of effective masses in these materials.

In an interesting session dealing with dendritic growth, Tiller, of Westinghouse, discussed at length the fundamentals of dendrite growth from theoretical considerations. In terms of surface energy, atomic kinetics, and other parameters, the solution of the differential equation for solute and temperature fields was presented.

Perhaps the most interesting session dealt with the growth of single crystals from the vapor phase. Wajda, of International Business Machines, presented an invited paper dealing primarily with the disproportionation of silicon iodide. This particular reaction is advantageous because it occurs at temperatures as low as 780°C; therefore, one can neglect solid-state diffusion phenomena. He discussed substrate properties, deposition temperatures, surface preparation of substrate, and the role which these parameters play in providing good epitaxial deposits. The various species that take part in the reaction and the specific mechanism involved in the growth of silicon layers were outlined in a flow-chart fashion. The role of iodine pressure, tube geometry, and thermal gradients was discussed. The use of doped material for a source in the reaction vessel was shown to give a substrate with the same doping level as the source material.

A direct deposition method of making p-n junctions and multilayer devices was reported by Allegretti, of Merck. As an illustration, he showed a multivibrator circuit made by the controlled doping of 11 layers of material. This paper invoked many enthusiastic comments and questions. Marinace, of International Business Machines, reported on the growth of germanium films by a closed-cycle germanium iodide reaction. He also discussed deposition on other crystal seeds, namely, gallium arsenide. He reported that the incorporation of iodine in these thin layers was orientation dependent.

It is clear that a technical conference on the metallurgy of semiconductors is a welcome forum for the presentation of papers by the materials people of the semiconductor industry. This has been demonstrated by the response to this year's meeting as well as that enjoyed at the 1959 meeting.

A similar meeting is planned for 1961. This meeting will be held in Los Angeles on Wednesday, August 30 through Friday, September 1, 1961. We are looking forward with anticipation to our third Technical Conference.

H. E. BRIDGERS is with the Bell Telephone Laboratories, Allentown, Pa.

SEMICONDUCTORS IN PRAGUE

A Report on the Fourth International Conference on Semiconductor Physics

by Harry C. Gatos, Lincoln Laboratory, MIT

The Fourth International Conference on Semiconductor Physics was held in Prague August 28 to September 2, 1960. The main topics covered were: band structure and related problems, transport phenomena, optical properties, resonance phenomena, radiation damage, excitons, photoconductivity, surface phenomena, thermal properties and thermoelectricity, ionic crystals, and semiconductor compounds.

It was a truly international conference attended by approximately 700 scientists representing many countries and all continents. Drs. Shockley, Bardeen and Brattain, the discoverers of the transistor which marked the new era in semiconductor physics were present. Of the approximately 260 papers presented, the majority came from American (110 papers) and Soviet (40 papers) laboratories.

Since the last international conference (1958), considerable progress has been made in all fronts of theoretical and experimental semiconductor physics. Transport phenomena received the greatest emphasis among the many fields of study in semiconductor physics (40 papers). As a result, the understanding of lattice scattering and electron-phonon interaction was markedly advanced. A distinctly new research area emerged in the last two years: carrier tunneling in narrow n-p junctions. This phenomenon was investigated recently by Esaki in Japan and led to the now well-known Esaki or tunnel diode which is an extremely fast switching, negative resistance element. Aside from its applied potential, this phenomenon provides a new approach to the study of important aspects of semiconductor physics. Significant results were reported on the effect of high magnetic fields (Lincoln Laboratory, M.I.T.) and on the effect of high pressures (Harvard University) on tunneling. Other significant reports on tunneling were from the General Electric Research Laboratory, Bell Telephone Laboratories, Hughes Products, and IBM Research Laboratory.

The presence of *helicons* in semiconductors was announced in a paper from the Ecole Normale Superieure (Paris). Helicons (circularly polarized waves) are present during the propagation of electromagnetic waves in a magnetic field. The helicons or their equivalent were recognized by plasma physicists.

There were a relatively small number of papers (9) on thermal properties and thermoelectricity. As it was pointed out by A. F. Joffe, a pioneer in thermoelectricity from the Academy of Sciences in Leningrad, there are still a number of unsolved problems regarding scattering of electrons and phonons and other aspects of thermoelectricity.

Surface phenomena continue to occupy an important place in semiconductor science and technology since communication with the semiconductor bulk properties must be achieved through surfaces. However, the preparation of *clean* and reproducible surfaces and their characterization remains a very complex problem. Progress is being made but at a relatively slow rate. Interesting papers were presented on the theory of surface states, surface diffusion effects, conduction phenomena, recombination, work damage, chemical interactions, etc.

Elemental semiconductors (silicon and germanium) are still the basic materials in semiconductor physics studies. Their extremely high purity in crystal imperfection, together with the broad experimental and theoretical knowledge of these materials makes them particularly suitable for truly-advanced and highly-refined studies. Nevertheless, many intensive and extensive studies have been undertaken in a large number of research laboratories toward the preparation and understanding of semiconducting compounds. There seems to be no doubt that new frontiers in semiconductor science and technology will be opened by semiconducting compounds. From the papers presented in this conference, it appears that III-V intermetallic compounds have received the greatest attention and are perhaps better understood than other classes of semiconductor compounds. In particular, a great deal of theoretical and experimental work was reported on InSb, GaAs, and GaSb.

As soon as compound semiconductors of suitable purity and perfection become available, they are promptly submitted to significant fundamental measurements. This became evident from the work reported on excitons in CdS and cyclotron resonance in CdAs$_2$. Because of their potential application to thermoelectricity, tellurides, sellenides, sulfides, and their alloys have been extensively investigated in the last two years in many countries. However, there were reports in other classes of materials. Organic semiconductors were studied in several laboratories. An interesting paper from the Academy of Sciences (Leningrad) dealt with glass-type semiconductors: chalcogenic glass exhibits purely electrical conductivity and shows the same effects and property changes as crystalline semiconductors.

Although a useful account of the many papers on semiconducting compounds cannot be given here, it became apparent during the conference that the unavailability of compounds of suitable purity and perfection is a more serious problem. Considerable progress by materials scientists on semiconducting compounds must be realized before the theoretical and technological potential of such materials can begin to be exploited.

Like other international conferences, this one afforded many scientists from a large number of laboratories a unique opportunity for communicating and exchanging ideas first hand. Such opportunities represent a significant factor in scientific progress. The proceedings of the Conference will be published by The Czechoslovak Academy of Sciences early in 1961.

ASTM CONSIDERS MATERIALS SCIENCES

or Atlantic City was Fine!

A Staff-Written Report

They came—some 3339 of them—to learn of the most recent advances in materials science. The mecca for their journey was the cool pleasant shore of Atlantic City— the purpose: the American Society for Testing Materials' (ASTM) 63rd Annual Meeting held from June 26 to July 1 at the Chalfonte-Haddon Hall.

The newly formed Materials Science Division sponsored the first days' activities which were composed of two related symposia: a morning session on recent progress in materials science and an afternoon session on the nature and origin of the strength of materials.

The luncheon speaker, Dr. W. O. Baker, vice president of research, Bell Telephone Laboratories, and a member of the President's Board of Consultants on Foreign Intelligence Activities, spoke on the national role of materials research and development. Dr. Baker emphasized the need for a national program of materials research and development, particularly applied to coordinating and combining the efforts of federal agencies, industry, and universities, in order to unify science and technology. This is being carried out, on a limited scale, through the cooperation of federal agencies which recently authorized a four-year program involving some $17-million granted to universities to aid in setting up interdisciplinary laboratories. Vast improvements in information handling services are also being made as part of this program.

Of the papers presented at the meeting, the following are of particular interest.

E. R. Parker, University of California, reported on the *Status of Ductile Ceramic Research*. Dr. Parker discussed the progress and summarized the results obtained in the development of ductile refractory ceramic materials with particular emphasis on dislocations in nonmetallic crystals, surface effects, sources of dislocations, and solution and precipitation hardening. Dr. Parker stated that, although there are some complications, dislocations in ionic crystals behave similarly to metal crystals. Magnesium oxide is one of the most promising materials being investigated because it is inherently ductile at room temperature. One of the advantages of studying ionic crystals is that relatively simple techniques can be applied to make dislocation networks visible. Studies employing these techniques have provided information which has been beneficial in explaining the dislocation mechanisms for both ionic and metal crystals.

J. J. Gilman, General Electric Co., presented a paper, *Dislocation Motion and the Yield Strengths of Solids*, which pointed out that the useful strength of many crystalline solids is limited by plastic yielding. Experimental observations indicate that the yielding is caused by the velocity of dislocation movement through the crystal. Dr. Gilman reported that yielding occurs at a sharply defined stress because the velocities at which the dislocations move vary directly with the applied stress. Although many factors have been found that influence dislocation velocities, some of the more important variables are: elastic modulus, dislocation type, glide plane, temperature, impurities, radiation damage, and plastic strain.

Resistance to Creep Deformation and Fracture in Metals and Alloys, by Frank Garafalo, U.S. Steel Corp., reviewed theories of creep with respect to available experimental findings. Void formation and intergranular fracture theories under creep conditions were discussed. The dependence of creep strength and fracture on structure was also discussed.

The topic, *Brittle Fracture and the Strength of Metals*, was presented by E. T. Wessel, Westinghouse Electric Corp. This paper emphasized that observed brittle-fracture strength of polycrystalline metals is not a unique basic material property, but rather, a gross product of interactions of several influential factors. The independent and composite effects of these factors were presented and discussed, and consideration was given to possible development of an understanding of the basic aspects of brittle fracture for practical application.

After each presentation, liberal time was alloted for discussion, and audience participation was invited.

Several AIME members were honored during the week-long activities. A. Allan Bates, a member of AIME since 1934, was elected to the one year presidency of ASTM. J. V. Emmons was elected to lifetime membership in recognition of his 50 years of continuous membership in ASTM. Also honored were Messrs. S. Umbreit and J. Vanick who were presented with Awards of Merit for outstanding service to ASTM.

While ASTM has been rapidly moving into the realm of materials science, we would hasten to add that The Metallurgical Society of AIME has not had its eyes closed to this growing field.

A review of the TRANSACTIONS over the years indicates that the society has devoted much time and effort to basic studies of materials and related processes. In recent years, however, even more emphasis has been placed on the science of materials and materials' fundamentals. Much of the work along these lines is being directed toward closing the gap between fundamental research and end-applications.

The October 1959 issue of the JOURNAL OF METALS was dedicated to materials engineering and metallurgical education. Among the topics discussed in the special issue were: future material problems, programs in materials engineering education, and industry's viewpoint on training in metallurgy and materials.

In addition, the Annual Fall Meetings of The Metallurgical Society for the past several years have contained numerous symposia and abstract sessions on materials science, and there are indications that future meetings will include even more presentations in this ever-expanding field.

AIME STAFF:
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ASST. SECRETARIES—J. B. ALFORD, H. N. APPLETON, J. C. FOX, R. W. SHEARMAN
ASST. TREASURER—JOHN LYNCH
METALLURGICAL SOCIETY STAFF:
SECRETARY—R. W. SHEARMAN
ASST. SECRETARY—O. C. JOHNSTON

NEWS OF YOUR PROFESSION

Southern Ohio NOHC Draws Record Attendance As Final Meeting Is Held in Columbus

The Southern Ohio Section of the National Open Hearth Steel Committee held its 24th and probably its last Annual Meeting in Columbus, Ohio, on September 23-24, at the Deshler-Hilton Hotel. The attendance set a new record with 217 registrants. In his opening remarks, William W. Bergmann, Chairman of the Section, announced that support of its activities was being withdrawn by one of the major steel companies in the area. The indication is that the interests of the Section can best be met by disbanding and having the members attend the meetings of neighboring sections in the future. Mr. Bergmann paid tribute to three groups whose participation made possible the success of the Southern Ohio Section of NOHC: companies, operating people, and suppliers. Mr. Bergmann is Superintendent, No. 1 Open Hearth, Armco Steel Corp., Middletown, Ohio.

The technical program was opened with a paper on *Safety With Oxygen* presented by George Dahm, Region Engineer, Linde Co., Cincinnati. It was pointed out that the increasing use of oxygen by the steel industry makes oxygen safety of great importance. Most accidents involving oxygen are caused either by a failure to understand the basic nature of oxygen, or to understand safety procedures. Necessary for combustion are three elements: oxygen, fuel, and a source of ignition. Elimination of any one will remove the hazard of combustion.

Production and Operating Problems With Roof Lance Furnaces was the subject of three technical papers. First speaker was W. O. Richmond, Open Hearth Superintendent, Armco Steel Corp., Ashland, Ky., who described the safety features of oxygen roof lances. If oxygen pressure drops, or if the pump fails, the lance automatically retracts, and the oxygen flow cuts off. If a serious water leak occurs, the water can be cut off at the panel board. Second speaker was Robert L. Sigl, Superintendent of Fuel and Power, Detroit Steel Corp., Portsmouth, Ohio. From the experience of one complete campaign and part of a second on two different furnaces, these factors were described as important in obtaining maximum production rates: thoroughly trained men; high fuel rates; use of fundamentally acceptable metallurgical practices; development of fast slag handling techniques; availability of high density scrap at all times; bridge walls of proper design; maintaining roof cleanliness for heat dissipation; cleaning of flue areas on a definite and routine schedule; avoidance of water in contact with the basic roof. Gerald Curtis, Fuel Engineer, Armco Steel Corp., Middletown, presented the third paper, stressing the essential need for low-cost oxygen. In July, 1959, a 130 tpd bulk oxygen plant went into service at Middletown. This has proved insufficient in capacity. An additional 140 tpd plant will be completed in

(*Continued on page 882*)

British Metals Group Will Meet in U. S. In Fall of 1961

The Iron and Steel Institute of Great Britain will hold a meeting in the United States and Canada in October and November, 1961. Hosts for the meeting will be The Metallurgical Society of AIME and the American Society for Metals. Dr. James B. Austin, vice president of research and technology, U.S. Steel Corp., is chairman of the Joint Society Committee.

The members of the British Iron and Steel Institute will arrive in New York on October 17. During their three-week stay, visits will be made to the Buffalo-Niagara Falls area, Cleveland, Pittsburgh, and Detroit, as well as a number of other steelmaking centers. During the visit a number of delegates will attend the annual Metal Congress, which will be held in Detroit the week of October 23.

The last meeting in the U.S. of the Iron and Steel Institute was held in 1910. A meeting planned for 1938 was cancelled because of unsettled world conditions.

Bar & Shaped Products Conference Scheduled Jan. 18 in Pittsburgh

Factors affecting the surface quality of bar products, and surface preparation of primary roll product will be among the subjects covered at the Conference on Bar and Shaped Products which will be held on January 18, 1961 at the Penn-Sheraton Hotel in Pittsburgh.

Sponsored by the Mechanical Working Committee of the Iron and Steel Division of The Metallurgical Society, the conference will be the third annual Conference on Mechanical Working of Steel. The two earlier conferences were devoted to flat rolled products.

Other subjects to be covered at technical sessions are double conversion and direct rolling; effects of composition, heating practice, pass design and other factors on the surface of bar products; and non-destructive testing. As with the two earlier conferences, a Proceedings volume will be published by Interscience Publishers, Inc., New York.

German Metallurgists Plan Celebration of 100th Anniversary

Dr. Howard Biers, senior consultant of the Market Research and Development Department of Union Carbide International Co., has been appointed to represent AIME at the 100th Anniversary celebration of the Verein Deutscher Eisenhuttenleute, a German ferrous metallurgy society. The Iron and Steel Division of The Metallurgical Society will be represented by William O. Binder, metallurgical consultant of Union Carbide Development Co. The anniversary celebration will be held in Dusseldorf Oct. 31-Nov. 4.

Foreign members have been especially invited to this year's annual meeting which will include three days of visits to important metallurgical installations beginning Oct. 31. A technical meeting will be held on Thursday, Nov. 3, followed by a membership meeting on Friday.

AROUND THE SECTIONS

Morenci (Arizona) subsection met on September 13 at the Longfellow Inn. After dinner the forty members present heard a talk by K. N. Woolen, protective atmospheres application specialist of General Electric Industrial Heating Department in Shelbyville, Ind. His subject was *Protecting Atmospheres and Furnace Equipment*, and was accompanied by slides showing equipment and installations for producing and employing protective atmospheres for purposes such as brazing and heat treating. The sub-section plans another dinner meeting on November 1, at which L. Klein of the Phelps Dodge Corp., will speak on the reforming of natural gas for reduction use in copper smelters.

El Paso section met September 14 at the Hotel Cortez. The members heard an address by Peter Puzzi, foundry superintendent of Darbyshire Steel Co. Mr. Puzzi spoke on *The Electric Furnace and New Developments in the Steel Making Industry.*

Lehigh Valley section made a fall field trip through the Grace Mine, Bethlehem Cornwall Corp., at Morgantown, Pa., on September 23. Seventy-seven members and guests were conducted on a tour of the underground operation and the surface installations, including the offices, shops, crushing plant, milling facilities, and tailings dam. The group also saw construction of pelletizing facilities which are scheduled for operation late this year. A buffet luncheon was served by the company. The section will hold a dinner meeting in late October.

Upper Peninsula section (Michigan) held its annual fall meeting on September 10. Following registration at the Michigan College of Mining and Technology at Houghton, members made a conducted tour through the Lake Linden area operations of Calumet & Hecla, Inc. A luncheon and a business meeting were held at the Calumet Elks Club. In the afternoon, members visited the Ahmeek Mining Area where they saw one of the largest operating steam hoists in the world. Cocktails and dinner were at the Main Lodge in Keweenaw Park. The dinner speaker was Jean Worth, editor of the Escanaba (Mich.) Press.

New York section held its September meeting at the Mining Club on September 29. Following dinner, there were two speakers on the *Future of Non-Ferrous Metals*. Irving Lipkowitz, assistant to the president of Reynolds Metals Co., discussed aluminum. Simon Strauss, vice president of American Smelting & Refining Co., discussed lead, zinc, and copper. Both talks were off-the-record.

Connecticut section held its September 21 meeting at the Chase Country Club in Wolcott. Following dinner there was a technical meeting at which Charles H. Moore spoke on *New Directions in the Copper Industry*. Dr. Moore is technical director of the Copper Product Development Association. He was formerly director of corporate research for P. R. Mallory Co.

Boston section, AIME, held its first meeting of the season on October 3. Eugene L. Olcott of Atlantic Research Corp., spoke on *Metallurgical Problems in Solid Propellent Rockets*. The November 7 meeting will feature a talk by John J. Bohrer of International Resistance Co., on *Properties of Metallic Thin Films*. At the meeting on December 5 (Richards Night), Robert M. Garrels of Harvard University will speak on *New Developments in Mineral Processing*.

San Francisco section's September 14 meeting at the Engineers' Club featured a talk and a film presented by E. R. Borcherdt of the Anaconda Co., on *Electric Wheel Haulage* at the company's Berkeley Pit. Peter Joralemon, chairman, presided at the meeting, which was attended by 76 members. The next meeting, which will be preceded by a dinner, will be held at the Engineers' Club on October 19.

Western section NOHC, has announced its tentative program for the coming year. At the October 19 meeting, R. C. Barrett and Ray Armstrong of Linde Air Div., discussed the *Use of Oxygen in the Steel Industry*.

The November 16 meeting will offer a talk by B. E. Etcheverry of Kaiser Steel Corp., on *The Growth Trend of the Western Steel Industry*. No meeting is scheduled for December.

At the January 18, 1961 meeting, a representative of Griffin Wheel Co., will discuss that company's pressure casting of railroad car wheels. John Golden and Howard Parker of Columbia Steel Div. will tell of their trip through the Far East and development of the steel industry in that area at the meeting on February 15. At the March 15 meeting, a representative of Bethlehem Steel Corp. will discuss the company's H-Iron process. *Production and Development of Certified Analysis Iron* will be the subject of a talk by an employee of National Supply Co., at the April 19 meeting.

Pittsburgh section will present an *Award of Merit Certificate* to J. H. Henderson, past chairman of the Section, at its Off-the-Record Meeting which will be held on November 4 at the Penn-Sheraton Hotel. The award is given as an acknowledgment of Mr. Henderson's distinguished and meritorious service, and as an appreciation for many contributions to the affairs of the Section and in recognition of his constructive leadership.

Magnetism Conference Scheduled Nov. 14-17 in New York City

The Sixth Annual Conference on Magnetism and Magnetic Materials will be held at the Hotel New Yorker in New York City, November 14-17. The conference is expected to attract more than 1000 engineers, physicists, chemists, and metallurgists to discuss fundamental and applied research in the field.

About 160 technical papers, covering all phases of magnetism, are included in the program. Ten invited papers will be read by scientists from the U.S., Japan, Holland, France, and England.

Among the topics to be discussed are the formation of thin films of magnetic materials and their application as memory units for computers, the study of magnetic resonance phenomena, magnetic ordering in helical spin systems, and the properties of magnets made of particles of extremely small size.

On the evening of November 16, the annual banquet of the conference will be held. The speaker will be Dr. T. Keith Glennan, head of the National Aeronautics and Space Administration, who will discuss some of the problems in the successful operation of space systems.

There will be an exhibit of new equipment, materials, and books.

Persons interested in attending the conference should write to Dr. L. R. Bickford, Jr., IBM Research Center, P. O. Box 218, Yorktown Heights, New York.

Directors of AIME Meet In Denver as SPE Holds Annual Fall Meeting

The Board of Directors of AIME held its fall meeting in Denver, Colorado, on October 5 at the new Denver-Hilton Hotel. President Joseph L. Gillson presided. The directors meeting took place during the 35th Annual Fall Meeting of the Society of Petroleum Engineers of AIME, which was held October 2-5. The petroleum engineers also served as host for the Board of Directors meeting of the Society of Mining Engineers on October 4, and their own board meeting on October 3. (The Metallurgical Society held its most recent Board of Directors meeting in Philadelphia on October 18 during the Society's Fall Meeting.)

The Society of Petroleum Engineers' fall meeting, which was the first to be held in the Rocky Mountain area, attracted some 3000 engineers and wives from the U.S. and foreign countries. Exhibits and technical sessions, which contained 74 technical papers on petroleum drilling and production, were held at the Denver City Auditorium.

At a Membership Luncheon on October 4, AIME President Gillson spoke on AIME affairs, and stressed the fact that the group is an example of a large organization with three constituent societies working together congenially. SPE president Wayne E. Glen of Continental Oil Co., presided at the luncheon.

A dinner meeting to honor President Gillson and the AIME Board of Directors was held by the Colorado Local Section on October 5 at the Denver Petroleum Club. Col. Wendell Fertig spoke on the *Romance of Mining*.

The Metallurgical Society was represented at the Denver meeting by Ronald R. McNaughton, President-elect of AIME; Carleton C. Long, President of The Metallurgical Society, and a director of AIME; J. Stuart Smart, Jr., President-elect of The Metallurgical Society, and a director of AIME; and James B. Austin and Richard C. Cole, directors of AIME.

Pritchard Is President of Iron & Steel Group

C. E. Pritchard has been elected president of the Association of Iron and Steel Engineers. Mr. Pritchard is general superintendent of rolling mills for Alan Wood Steel Co. H. L. Jenter of U.S. Steel Corp., was elected first vice president, and C. W. Bruce of Republic Steel Corp., is second vice president.

Other officers elected include H. R. Knust, treasurer, and N. C. Michels, secretary.

William J. Schlitt

William J. Schlitt Named to Receive Reinartz Scholarship

William Joseph Schlitt III has been named to receive the Leo F. Reinartz Scholarship Award for 1960-61. The award, sponsored by the National Open Hearth Steel Committee of the Metallurgical Society's Iron and Steel Division, is given to an outstanding student who elects to study metallurgy at Carnegie Institute of Technology, Mr. Reinartz' alma mater. The scholarship provides a $750 stipend.

Selection of the award winner is made by the Leo F. Reinartz Scholarship Award Committee. Mr. Schlitt graduated first in his class of 250 from the Thomas Jefferson High School in Clairton, Pa. He was president of the local chapter of the National Honor Society, and was picked to participate in the Science Honors Institute, and the American Chemical Society Examination. He was a semi-finalist is National Honor Society Competition for scholarships. Mr. Schlitt is enrolled in his first semester at Carnegie Tech.

The Leo F. Reinartz NOHC scholarship was first awarded in 1955. It is endowed with a $20,000 fund established by the National Open Hearth Steel Committee to honor Mr. Reinartz, 1954 President of AIME, for his many years of work with NOHC. Mr. Reinartz is a consultant with the Armco Steel Corp., Middletown, Ohio.

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Metallurgical Society Publishes Volume VII on Nuclear Metallurgy

The Metallurgical Society has announced its publication of *Nuclear Metallurgy, Volume VII*. This is the tenth in a special report series of the Society's Institute of Metals Division.

Covering discussions on metallic moderators and cladding materials, the new volume includes technical papers presented at the Fall Meeting of the Society, which was held in Philadelphia, October 17-20. The papers were read at the seventh symposium sponsored by the Nuclear Metallurgy Committee of IMD.

In outlining the symposium theme, as represented in the new book, Conference Chairman Donald E. Thomas, of Westinghouse Astronuclear Laboratory said, "The committee has traditionally attempted to select symposium topics of maximum current interest in order to stimulate activity and discussion on timely nuclear material problems.

"Although the early production reactors utilized solid graphite moderator, requirements for more compact solid moderator material serviceable at higher temperature than is feasible with light and heavy water have led to consideration of metal hydrides. The alloys of zirconium and yttrium with hydrogen have particular merit as solid moderators, and the present volume contains information on the physical and mechanical properties of these materials.

"The portion of the symposium dealing with cladding materials relates recent experience with the fabrication and properties of the commercially established material *Zircaloy 2*, and the tendency for this material to pick up hydrogen in service. Recent developments in the search for zirconium base alloys having improved high-temperature strength or decreased tendency for hydrogen pickup in high-temperature irons, nickels, and chromium of particle -strengthened aluminum and in high-temperature irons, nickels, and chromium-base alloys is also reported."

Contents of the volume are as follows:

Fabrication and Evaluation of Zircaloy-2 and Zircaloy-4 Tubing Used to Clad Uranium Dioxide and Aluminum-Plutonium Fuel, by R. C. Aungst, K. M. Haws, and P. L. Farnsworth.

Mechanical Behavior of Cold Worked Nuclear Grade Zircaloy-2 Tubing, by H. H. Klepfer and C. N. Spalaris.

(*Continued on next page*)

Thermoelectric Energy Conference Slated For Jan. 1961

The Metallurgical Society will be one of the participating Societies at a *Symposium on Thermoelectric Energy Conversion* which will be held at the Statler-Hilton Hotel in Dallas, Texas, on January 8-12, 1961.

Because of the interdisciplinary nature of the technical problems associated with direct energy conversion, the meeting is designed to provide communication among members of several technical societies and between industrial, university, and military programs. The program will be supplied and conducted by the participating societies.

The program will cover such subjects as related aspects of direct conversion; physical theory; synthesis and evaluation of thermoelectric materials; environmental effects; materials fabrication and assembly; methods of measurement; design parameters; and device construction and performance.

It is intended that the proceedings of the conference will be published, either as a book or as a supplement to a technical journal. Papers will be included in the published proceedings by agreement of the author and the editors.

In addition to The Metallurgical Society, which is represented by Dr. Arthur J. Rosenburg, Tyco, Inc., participating societies include the American Institute of Electrical Engineers; American Nuclear Society; American Rocket Society; American Society of Heating, Refrigerating and Air Conditioning Engineers; the Electrochemical Society; and the Institute of Radio Engineers. The U.S. Department of Defense is acting as a cosponsor.

Requests for hotel rooms during the convention should be sent directly to the Dallas Statler-Hilton. Late registrants will be assigned to the Sheraton-Dallas or to other hotels in the area.

Nuclear Metallurgy

(Continued from previous page)

Creep and Corrosion Behavior of Some High Strength Zirconium Alloys, by D. L. Douglass.

Zirconium Alloys for Cladding High Temperature Fuel Elements, by R. K. Wagner, H. E. Kline, and D. I. Sinizer.

Fabrication and Mechanical Properties of Aluminum Powder Metallurgy Products, by E. G. Kendall and W. H. Friske.

A Review of Metal Hydrides for Nuclear Reactor Applications, by W. M. Mueller and J. P. Blackledge.

Physical Properties of Yttrium Hydride, by E. S. Funston.

The Zr-H System, by J. B. Vetrano and D. F. Atkins.

Mechanical Properties of Zirconium Hydride, by W. M. Mueller and R. L. Beck.

Compatibility of Zirconium Hydride and Carbon Dioxide, by H. E. Shoemaker.

Cladding of Delta-Phase Zirconium Hydride, by E. S. Hodge and S. J. Paprocki.

The price of the volume is $5.00 to members and $7.00 to non-members. It may be obtained at the registration desk during The Fall Meeting or from the American Institute of Mining, Metallurgical, and Petroleum Engineers, 29 West 39th Street, New York 18, N. Y.

ASTME Plans Seminars On Manufacturing Techniques & Processes

The American Society of Tool and Manufacturing Engineers (ASTME) is sponsoring a series of Creative Manufacturing Seminars designed to offer information on the manufacturing techniques, ideas, and processes being utilized by industrialists throughout the country.

The registration fee for each seminar is $45 for members of ASTME and $60 for non-members. The fee includes all Seminar sessions, two luncheons, and copies of technical papers and research reports presented. Persons interested in attending any of the seminars should contact Gilbert E. Seeley, Education Director, ASTME Headquarters, 10700 Puritan, Detroit 38, Mich.

Price Will Increase For Transactions

Announcement has been made by The Metallurgical Society that effective January 1, 1961, the subscription price of the bi-monthly TRANSACTIONS OF THE METALLURGICAL SOCIETY OF AIME, will be increased for non-members from $20.00 per year to $30.00 per year. For non-members outside of the Western Hemisphere, a subscription will cost $32.00. These increases are necessitated by increased publication costs. There will be no increase of the yearly subscription to members, remaining at $5.00 per year.

Bound volumes, if ordered when paying annual dues will remain at $5.00 for members. If ordered later, the charge for bound volumes of TRANSACTIONS will be $6.50. Bound volumes for non-members will cost $35.00 after January 1, 1961.

Because of the expense of carrying bound volumes of back issues in inventory, the charge for back volumes will be increased to $35.00 for non-members on January 1, with 30 pct discount being given to members, or $24.50 per volume.

Members may wish to purchase copies of bound volumes now before the price increase becomes effective. The following price schedule for members will exist for the balance of the year for those volumes that remain in stock:

1958-1959
$5.00 per copy (non-members, $25.00)

1954-1957
$6.30 per copy (non-members, $9.00)

1953
$4.90 per copy (non-members, $7.00)

Metals Branch TRANSACTIONS for the years 1949-1952 inclusive are currently out of stock. Arrangements have been made with Johnson Reprint Corp. to reprint Volumes 1-4 of JOURNAL OF METALS, including TRANSACTIONS. A discount price will be available for members of AIME. This will make it possible for individuals and libraries to obtain a complete set of Metals TRANSACTIONS from 1949 to the present. Orders for the years 1949-1952 should be sent to Johnson Reprint Corp., 111 Fifth Avenue, New York 3, N. Y.

Southwestern NOHC Holds Fall Meeting In Texas, Oct. 13-14

The Southwestern section, NOHC, held its annual fall meeting Oct. 13-14 in Texas. Following a plant visit to the E. B. Germany Works of Lone Star Steel Co., on Thursday, Oct. 13, the group went by train to Dallas, where, on Oct. 14, technical sessions were held at the Sheraton-Dallas Hotel.

The technical program included a symposium on *Carbon Additives*, with Simon Fredericks presiding, and talks on *Experience in the Production of High, Medium, and Low Carbon Steels By the Basic Oxygen Process* by C. C. Benton; *Basic Problems and Results in the Use of Oxygen* by Leonard Pucker and Ed Balog; *New Developments in Hot Topping Practice* by Harold Oldan, H. W. Patlogar, and J. S. Smith; *The Potential of the Open Hearth* by George Ferris; *The Vacuum Spectrograph as a Production Tool* by W. R. Thorpe; and *Open Hearth Basic Pre-Fab Sections (masonry)* by L. R. Lattimore, A. H. Sommer, and L. B. Lindemuth.

A Fellowship Dinner was held on Friday evening, Oct. 14. The speaker was Robert Murphy, district attorney, of Nacogdoches, Tex.

Personals

(Continued from page 824)

Development Association. He has served as vice-chairman of the Institute of Metals Division of AIME.

C. W. Schwenzfeier has been appointed vice president and general manager of the Elmore Division of Brush Beryllium Co. **W. W. Beaver** has been appointed vice president of research and development.

H. J. Pfeifer has been appointed sales manager of the Phillipsburg district for Union Carbide Metals Co. **O. G. Specht, Jr.,** has been named manager of the Pittsburgh sales region, and **A. Herbener** has been named manager of the Pittsburgh sales district.

George J. Peer has been named sales manager, furnace products, of Basic, Inc., Cleveland, Ohio. **D. Bedell Baxter** is the new sales manager for the company's central district in Cleveland.

Paul G. Sammet has been named assistant general superintendent in charge of melting operations at Ashland works of Armco Steel Corp.

W. E. Mooz has joined U.S. Borax & Chemical Corp. of Los Angeles. He was formerly assistant to the plant manager of Titanium Metals Corp. of America.

E. V. Blackmun, assistant chief metallurgist, die casting division, Aluminum Co. of America, has received the annual Doehler Award, the highest honor of the die casting industry. The award was established in 1949 by Doehler-Jarvis Corp., a division of National Lead Co., to recognize outstanding contributions to the advancement of the die casting art.

Thomas Cleary, Jr. has been promoted to assistant general superintendent of rolled and tubular products of the Youngstown Sheet and Tube Co. He was formerly open hearth superintendent.

Marshall B. Nunlist is now vice president, finance and administration, of the Brush Beryllium Co. He was formerly vice president and treasurer of R. Hoe & Co., Inc.

Seven past presidents of the Metal Powder Industries Federation were honored by that organization at a banquet on Sept. 17. The current president, **George A. Roberts,** presented engraved plaques to: **Harold E. Hall** (posthumously), Metals Disintegrating Co.; **Biderman T. du Pont,** Crane Co.; **Thomas L. Robinson,** Powdercraft Corp.; **Ernest H. Klein,** New Jersey Zinc Co.; **Thomas H. Moore,** Antara Chemicals div., GAF; **Paul E. Weingart,** American Metal Climax, Inc.; and **Morris Boorky,** Presmet Corp.

Books

(Continued from page 832)

and instrumentation and refractories in steel making plants. Written by a British author connected with Banaras University in India, the book draws on international sources for material, illustrations, and examples. •

Crystal Chemistry of Simple Compounds of Uranium, Thorium, Plutonium, and Neptunium, by E. S. Makarov, *Consultants Bureau Inc.*, New York, 142 pp., $5.25, 1960—This is said to be the first attempt to correlate crystal structure data on the simplest compounds of the most thoroughly studied actinide elements. No books dealing specifically with crystal chemistry of the actinides have been published previously, although separate surveys have appeared in various volumes. •

Welding Handbook, fourth edition, Part III: Special Processes and Cutting, edited by Arthur L. Phillips, Cleaver-Hume Press Ltd., 500 pp., about $10.00, 1960.—This third section deals with many of the newest and fastest developing aspects of welding; including chapters on *Welding of Plastics, Adhesive Bonding of Metals, Ultrasonic Welding,* and *Welding by Cold Working.* It also covers in detail the established processes of thermit welding, induction welding, brazing and soldering, the surfacing of metals, and the process of metalizing. •

Symposium on Newer Metals, ASTM Special Technical Publication No. 272, 218 pp., $7.25, 1960—This book discusses the needs for materials with properties superior to those of existing materials for new applications in aircraft, nuclear energy, and communications. The book is illustrated and covers three distinct topics: *Properties of Refractory Metals; Nuclear and Light Metals;* and *Processing of Newer Metals.*

The first section includes: *Universal High-Temperature Testing Machine for Vacuum or Controlled Atmosphere* by M. J. Manjoine, L. L. France, and R. T. Begley; *Mechanical Properties of Molybdenum and Molybdenum-Base Alloy Sheet* by M. Semchyshen and Robert Q. Barr; *High Temperature Mechanical Properties of Tantalum* by F. C. Holden, F. R. Schwartzberg, and R. I. Jaffee; *Effect of Oxygen and Nitrogen on Workability and Mechanical Properties of Columbium* by R. T. Begley and L. L. France; and *Tensile Properties of the Platinum-Group Metals* by F. C. Holden, R. W. Douglass, and R. I. Jaffee.

The section on *Nuclear and Light Metals* includes *Effect of Purity and Manufacturing Variables on Elevated*

(Continued on page 883)

Authoritative books for engineers

NONDESTRUCTIVE TESTING HANDBOOK

This pioneering Handbook presents all the means of testing the essential properties and performance capabilities of materials, parts, components, and structures *without impairing serviceability.*

Explains and illustrates applications, advantages, limitations of 24 different test methods applicable in every branch of engineering and industry. Specifications, equipment costs, hundreds of pages of test interpretation data. *Prepared under the auspices of the Society for Nondestructive Testing. 1,100 illustrations, graphs, tables; 1,910 pp. 106 Contributing, Consulting Editors. Robert C. McMaster, Ed. October, 1959.* 2 volumes, $24

PRODUCTION HANDBOOK

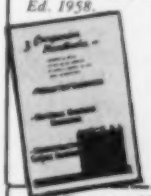
Packed with practical production know-how, this Handbook is the complete key to management-engineering methods that are revolutionizing industrial production today. Fully covers new materials, machines, processes, and proper functioning of company organization.

Supplies proved principles, time- and work-saving systems, and successful operating procedures for maximum productivity at minimum cost. Saves hours of research and costly experimentation. *725 illustrations, tables; 1,726 pp. 48 Contributing, Consulting Editors. Gordon B. Carson, Ed. 2nd Ed., 1958.* $16

MATERIALS HANDLING HANDBOOK

The complete guide to modern materials handling. This unique Handbook explains the governing principles, today's most efficient methods and systems, and recommended equipment for moving material at least cost — whether in raw, in-process, or finished form.

Useful in all industrial situations. Handbook answers every basic question of work flow within and outside the plant. *Sponsored by The American Society of Mechanical Engineers and the American Material Handling Society. 991 illustrations, tables; 1,740 pp. 84 Contributing, Consulting Editors. Harold A. Bolz, Ed.; George E. Hagemann, Assoc. Ed. 1958.* $20



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Southern Ohio Section NOHC. . . .

(Continued from page 877)

the spring of 1961. Storage capacity will total 260,000 cu. ft.

Bath Temperature Control on Oxygen Lance Furnaces was described by Thomas D. Moore, Armco Steel Corp., Middletown. Accelerated oxidation rates take their toll of roof refractories. Through the use of expendable thermocouples, sufficient data were developed to plot a minimum pouring temperature line. The use of a bath temperature control chart began on May 1, 1960 and resulted in a reduction of fuel consumption and an increase in oxygen flow rates. This was the award winning paper of the Southern Ohio Section; the author was honored with a check for $100.00.

A Method of Nozzle Setting was explained by W. O. Richmond of Armco. Through the use of ladle well blocks, these advantages have been realized: availability of ladles has been increased; a hot and hazardous job has been eliminated; ladles are back on the job promptly.

The final morning paper was presented on *Ladle Gunning* by Donald Saylor, General Foreman, Masonry Dept., Ford Motor Co., Dearborn, Mich. With 14 ladles to serve 10 open hearth furnaces, the demand for ladles increased as tap-to-tap time decreased; hence, the introduction of ladle gunning which has added five or more heats to the brick life. A second gunning is possible. The results of 39 gunned linings are: 37 pct increase in lining life, 18 pct decrease in labor, and 15 pct decrease in materials. Another advantage is the easier removal of skulls on gun-lined ladles.

Serving as Chairmen were R. K. Thornberry of Acme-Newport Steel Co., and McBeth Sample, General Foreman, No. 2 Open Hearth, Armco Steel Corp., Middletown.

Leadoff paper on the afternoon session was *Quality of Aluminum Killed Steel Ingots* by O. V. Ireland, Assistant Works Metallurgist, Armco Steel Corp., Middletown. Aluminum was added after the ferrosilicon block, either all in the ladle, or a split technique with half in the ladle and half in the molds. Ingot surfaces were studied after shot blasting, which removed oxides, but not steel. A second paper on the same subject was presented by John Nordstrom, Superintendent of Quality Control, Detroit Steel Corp., Portsmouth. He stressed the importance of good practice to obtain good results: keeping undesirable elements out of charged material; using good scrap of high density and high purity; maintaining bath temperature at 2850° to 2880° F; and preparing molds and stools properly.

Pneumatic Injection of Graphite in Open Hearth Bath was the subject of a paper by R. K. Thornberry of Acme-Newport Steel Co. In his cold iron shop, there is a need to conserve pig iron. For pneumatic injection of graphite, a steel pipe, 10 ft long, and 1 in. in diameter is used. The end of the pipe is placed 4 in. below the molten steel surface. The pipe is kept as nearly vertical as possible to prevent graphite from reaching the slag. This has proved a useful tool for soft melting heats—those not hot enough to tap where carbon injection promotes a boil, thereby raising the temperature; and those heats melting in with carbon content too low to meet specifications.

A system of *Removal of Slag and Refuse From Open Hearth Pits* was described by Don McCormick, Armco Steel Corp., Middletown. Faster heat times have made slag removal a more acute problem—both in the volume to be removed, and the more limited time to remove slag between heats. One furnace has been equipped in the pits with a concrete ramp down which the slag can drain. A bulldozer is used to clean up the ramp, and slag is removed with a truck. Advantages are: increase in safety; overhead crane is 17.4 pct more available; labor in the pit is more available; and engine switching time is decreased.

The technical program ended with *Progress Reports on Basic Roofs*. Reports on their respective plants were given by C. Hesselman, Masonry Superintendent, Acme-Newport Steel Co.; Kenneth Ott, General Foreman, Open Hearth, Detroit Steel Corp.; and L. Yoder, General Foreman, Masonry Department, Armco Steel Corp., Middletown.

Chairmen of the afternoon session were O. V. Ireland of Armco, and Eugene Allison, Superintendent of Masonry, Detroit Steel Corp.

Highlight of the Conference was the Annual Dinner, with an address by the Rev. Mr. Laurence Hall, Rector of St. Paul's Episcopal Church, East Cleveland, Ohio. Speaking on *The High Cost of Laughing*, Mr. Hall prescribed laughter for its therapeutic effect in combating disease. Mr. Hall asked the audience this question, "Can you laugh at yourself?" This is a key to successful living. Serving as Toastmaster was the Chairman of the National Open Hearth Steel Committee, Lester W. Moore, Assistant General Manager, Wheeling Steel Corp., Steubenville, Ohio.

The Conference concluded on Saturday with many registrants attending the Ohio State vs Southern Methodist football game. Also on Saturday, a meeting of the Executive Board of the National Open Hearth Steel Committee was held.

MEMBERSHIP

Proposed for Membership
Metallurgical Society of AIME

Total AIME members on September 30, 1960, was 34,343; in addition 2,562 Student Members were enrolled.

ADMISSIONS COMMITTEE

W. L. Brytczuk, Chairman; S. C. Carapella, Jr.; J. W. Hanley; T. D. Jones; H. C. Larson; Harold Margolin; Shadburn Marshall.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

Members

Bean, Charles H., Argonne, Ill.
Cummins, Herbert H., East Chicago, Ind.
Ellis, Frank J., Montreal, Que., Canada
Fisch, Herbert A., Schenectady, N. Y.
Gross, Alfred G., Jr., Avon Lake, Ohio
Heller, Enrique, La Oroya, Peru
Howe, Rayworth F., Butte, Mont.
Kowall, Nicholas, Williamsville, N. Y.
Hrysko, Wladimir D., Kensington, Sydney, Australia
Lee, Louis T., East Chicago, Ind.
Mark, Albert, Asbury Park, N. J.
Mumper, Thomas O., Newton Square, Pa.
Pearson, John, China Lake, Calif.
Peterson, Norman M., Mountain View, Calif.
Plessinger, Clyde B., Lindale, Tex.
Powers, Glenn F., Monroe, Tex.
Rosenberg, Arthur J., Boston, Mass.
Snyder, Henry M., Jr., Millville, W. Va.
Templin, James P., Palmyra, N. J.
Totten, Warren L., Somerville, N. J.
Tuten, Harold R., Beverly, Ohio
Willenbrock, H. Carl, Jr., Pittsburgh, Pa.
Woodbury, Roger K., Los Angeles, Calif.

Associate Members

Blair, John, Cambridge, Mass.
Cabell, Richard A., New York, N. Y.
Mack, Stanley J., Chicago, Ill.
Pilkanis, Francis V., Phillipsdale, R. I.
Wieland, J. Lee, Hinesdale, Ill.

Junior Members

Braun, Harvey D., New York, N. Y.
Fenbert, Henry R., Dearborn, Mich.
Grote, Richard E., Mahwah, N. J.
Jordan, Eugene F., Stow, Mass.
Kephart, Alan R., Scotia, N. Y.
Mathis, Donald L., Rolla, Mo.
Mazzarella, Vincente, Sao Paulo, Brazil
Orrok, George T., Murray Hill, N. J.
Schummer, Arthur D., Luxembourg
Spitzig, William A., Cleveland, Ohio

REINSTATEMENT—CHANGE OF STATUS

Associate to Member

McMillen, Richard L., Sahagun, Mexico

Junior to Member

Brooks, Allan L., Long Island City, N. Y.
Hansen, Barry J., El Salvador, Chile

Student to Member

Arentzen, Charles, Anaconda, Mont.
Kablash, Thomas L., Pittsburgh, Pa.
Riffin, Paul V., Lexington, Mass.

Student to Associate

Guimaraes, Helio P., Belo Horizonte, Brazil

SPECIAL REINSTATEMENT—MEMBER

Gibson, Francis E., Lansing, Ill.

Junior Member

Kovach, Dennis M., Port Huron, Mich.

Student Member

McMahon, Michael J., III, Chicago, Ill.

STUDENTS

Anderson, James H., Jr., Palo Alto, Calif.
Heacox, Richard A., University Park, Pa.
Khangaonkar, Padmakar R., Troy, N. Y.
Lorkovic, William M., Norman, Okla.
Proske, Joseph W., DuBois, Pa.
Quets, Jean, Salt Lake City, Utah
Smeal, Charles R., Altoona, Pa.

CHANGE OF STATUS

Junior Reverting to Student

Mular, Andrew L., Mattapan, Mass.

Education

(*Continued from page 840*)

strength, and stress to produce one pct creep strain or rupture in designated times are presented in 64 figures. Data are given for 28 alloys in sheet form. An extensive bibliography contains reference to 121 reports and articles pertaining to test methods and equipment and to very-short-time data for these and many other materials. **Order OTS PB 151088.**

DMIC Report 132, *Design Information on Nickel-Base Alloys for Aircraft and Missiles.* Tensile, compressive, shear, and bearing properties of some nickel-base alloys for aircraft and missiles have been assembled and evaluated. Background information on these alloys is presented to aid in the selection of alloys for given elevated-temperature applications. Order **OTS PB 151090.**

DMIC Report 133, *Tantalum and Tantalum Alloys.* A descriptive literature survey has been made covering physical properties, process metallurgy, binary and ternary alloy systems, chemical properties, and metallurgical properties of tantalum and tantalum alloys. The properties of tantalum are well reported, but investigations concerned with the effects of interstitial and substitutional alloying elements on the properties of tantalum are limited. Order **OTS PB 151091.**

DMIC Report 134, *Strain Aging of Refractory Metals.* The available data on strain-aging behavior of 12 refractory metals (vanadium, columbium, tantalum, chromium, molybdenum, tungsten, ruthenium, rhodium, palladium, osmium, iridium, and platinum) are reviewed and interpreted. The body-centered-cubic metals, tantalum, columbium, and vanadium, tend to exhibit greater strain-aging effects and lower transition temperature than do tungsten, molybdenum, and chromium. Available data indicate that strain aging is a result of the interaction of dislocations with oxygen and carbon in tantalum, oxygen and possibly hydrogen in columbium, carbon in molybdenum, and nitrogen in chromium. The interstitial solute atoms responsible for the strain aging of vanadium and tungsten are unknown.

Face centered cubic metals (rhodium, palladium, iridium, and platinum), and hexagonal close packed metals (ruthenium and osmium) exhibit strain-aging effects similar to those observed in the two groups of body centered cubic refractory metals. However, the available data do not permit the determination of the identity of the substitutional solute atoms causing the strain-aging effects. Order **OTS PB 151092.**

NECROLOGY

Year Elected	Name	Date of Death
1919	Brooks, Raymond	Aug. 12, 1960
1916	Crawford, William E.	Unknown
1957	Erlandson, Roy M.	Aug. 31, 1960
1925	Fletcher, J. H.	July 18, 1960
1931	Goldsmith, Marc S.	May 2, 1960
1940	Harrison, S. T.	July 30, 1960
1937	Ihrig, Harry K.	Aug. 22, 1960
1952	Johnson, Stanley W.	Mar. 25, 1960
1943	Plews, George	Aug. 13, 1960
1931	Schumacher, W. P.	Aug. 11, 1960
1917	Uren, Lester C.	Aug. 21, 1960

Books

(*Continued from page 881*)

Temperature Properties of Beryllium by W. W. Beaver, R. G. O'Rourke, and J. N. Hurd; *Stress-Rupture and Creep Properties of QMV Beryllium Metal* by the same authors; *Test Methods Used for Zirconium and Zirconium Alloy in the Naval Reactor Program* by Harold L. Kall; *Effects of Heat Treatments on the Tensile and Corrosion Properties of Zircaloy 2* by J. G. Goodwin, L. S. Rubenstein and F. L. Shubert; *Survey of the Mechanical Properties of Yttrium and Yttrium Alloys* by O. N. Carlson, D. W. Bare, E. D. Gibson, and F. A. Schmidt.

Processing of Newer Metals includes *Columbium and Tantalum Fabrication* by H. M. McCullough; *Deoxidation and Denitrification Studies of Chromium and the Effect on Low-Temperature Properties* by H. L. Levingston and N. J. Grant; *Review—the Fabrication of Tungsten* by C. H. Toensing; *Solubility of Nitrogen and Oxygen in Lithium and Methods of Lithium Purification* by E. E. Hoffman; and *Coulometric Determination of Tin with Electrolytically Generated Iodine: Application to Analysis of Zircaloy* by Roy D. Caton, Jr., and Harry Freund.

•

Annual Engineering Management Meeting Held in Chicago

The eighth annual Engineering Management Conference was held at the Morrison Hotel in Chicago, Sept. 15-16. Technical sessions covered such subjects as *Impact of Technological Progress on Tomorrow's Management, Statistics as an Aid to Engineering,* and *Social Change in Science and Engineering.* A banquet was held on Thursday evening, Sept. 15. The speaker was Peter F. Drucker, professor of management at the Graduate Business School of New York University.

Letters

(*Continued from page 828*)

three was reversed somewhere in transcription.

In describing the force delivered by the Impacter, the expression 15,000 lb per blow was used. When delivering a force to the material, it should be described in terms of ft-lb. The actual force exerted by the Impacter is *15,000 ft-lb per blow.*

It is understood that the comments made here can be looked upon as being minor in nature, but nevertheless, misinterpretation by readers could result. We would appreciate you correcting these errors, if at all possible.

C. P. Mueller
Technical Manager
Refractomet Division
Universal-Cyclops
Steel Corp.

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Coming Events

Nov. 4, Pittsburgh Section, AIME, 15th Annual Off-the-Record Meeting, Penn-Sheraton Hotel, Pittsburgh.

Nov. 8-11, First National Die Casting Exposition and Congress, Detroit Artillery Armory, Detroit.

Nov. 14-16, Fifteenth Technical and Operating Conference, Steel Founders' Society of America, Hotel Carter, Cleveland.

Nov. 14-17, Sixth Annual Conference on Magnetism and Magnetic Materials, Hotel New Yorker, New York City.

Nov. 15, Buffalo Section, NOHC, 11th Annual Fall Meeting, Statler-Hilton Hotel, Buffalo.

Nov. 16, Annual Award Meeting, Niagara Frontier Section of AIME, Hotel Stuyvesant, Buffalo.

Nov. 16, Western Section, NOHC, Roger Young Auditorium, Los Angeles.

Nov. 16, Cleveland Section, NOHC, The Saddle Inn, Avon Lake, Ohio.

Nov. 16, Powder Metallurgy Group, New York Section, AIME, Brass Rail Restaurant, 43rd and Fifth Ave., New York City.

Nov. 30-Dec. 2, AIME 18th Electric Furnace Conference, Morrison Hotel, Chicago.

Jan. 8-12, 1961, Symposium on Thermoelectric Energy Conversion, Statler-Hilton Hotel, Dallas, Texas.

Jan. 18, 1961, Conference on Bar and Shaped Products, sponsored by Mechanical Working Committee of The Metallurgical Society, Penn-Sheraton Hotel, Pittsburgh.

Feb. 26-March 2, 1961, AIME Annual Meeting, Ambassador and Chase & Park Plaza Hotels, St. Louis, Mo.

Mar. 13-14, 1961, 59th Annual Meeting, Steel Founders' Society of America, Drake Hotel, Chicago.

Apr. 10-12, 1961, 44th National Open Hearth Steel Conference and Blast Furnace, Coke Oven, and Raw Materials Conference, Sheraton Hotel, Philadelphia.

Apr. 12-14, 1961, AIME International Symposium on Agglomeration, Sheraton Hotel, Philadelphia.

Apr. 26-27, 1961, Cleveland Section and The Metallurgical Society, Technical Conference on High Temperature Materials, Hotel Carter, Cleveland.

May 18-19, 1961, AIME 15th New England Regional Conference, Hotel Van Curler, Schenectady, N. Y.

Oct. 17-Nov. 3, 1961, Visit of the Iron & Steel Institute (Great Britain) to the United States.

Oct. 23-26, 1961, Fall Meeting of The Metallurgical Society of AIME, Pick-Fort Shelby Hotel, Detroit.

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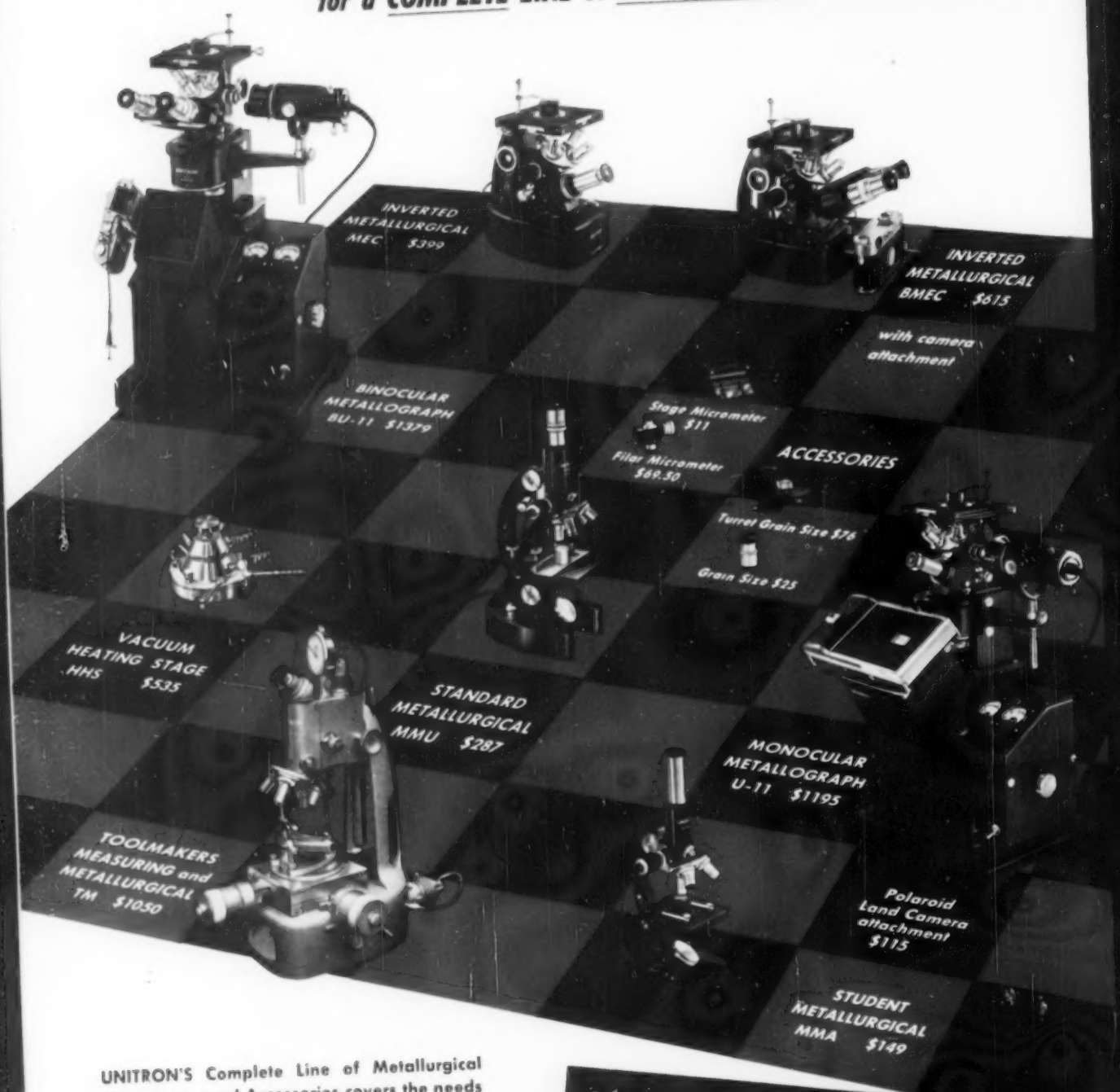

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